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DETERMINING THE EFFECTIVENESS OF AN AFTERBURNER TO REDUCE DIOXINS AND FURANS

by

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16. ABSTRACT During reactivation of granular activated carbon (GAC) used in the treatment of municipal water supplies, a potential exists for the release of adsorbed organic compounds or their reaction products into the environment. The purpose of this study was to evaluate the effectiveness of an afterburner in reducing or eliminating the occurrence of dioxins and furans in the reactivator effluent streams. Quantitative measurements were made of tetrachlorodibenzo-p-dioxins (TCDDs) and tetrachlorodibenzofurans (TCDFs) in all effluent streams from the Cincinnati, Ohio Water Works, reactivator unit and penta-, hexa-, and octa-chlorinated dioxins (CDDs) and furans (CDFs) in a preselected number of samples. During two of the tests, the natural gas and fuel oil were sampled to determine if PCBs were present in the fuel supply used to fire the carbon reactivation system.

No 2,3,7,8-TCDD was detected in any emission or process streams. TCDD was not detected in any stack particulate samples; however, small concentrations were detected in the vapor phase of the afterburner and recuperator. Low concentrations were also found in one cyclone catch sample. Octa-CDD and hepta-CDD were detected in all emission streams.

No 2,3,7,8-TCDF was detected in the particulate phase of any emmision stream, however, trace amounts were found in the gas phase of all emission streams. Low concentrations were also observed in one cyclone sample. One one PCB isomer was detected in the natural gas supply and none in the fuel oil.

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FOREWORD

The U.S. Environmental Protection Agency is charged by Congress with protecting the Nation's land, air, and water systems. Under a mandate of national environmental laws, the agency strives to formulate and implement actions leading to a compatible balance between human activities and the ability of natural systems to support and nurture life. The Clean Water Act, the Safe Drinking Water Act, and the Toxic Substances Control Act are three of the major congressional laws that provide the framework for restoring and maintaining the integrity of our Nation's water, for preserving and enhancing the water we drink, and for protecting the environment from toxic substances. These laws direct the EPA to perform research to define our environmental problems, measure the impacts, and search for solutions.

The Water Engineering Research Laboratory is that component of EPA's Research and Development program concerned with preventing, treating, and managing municipal and industrial wastewater discharges; establishing practices to control and remove contaminants from drinking water and to prevent its deterioration during storage and distribution; and assessing the nature and controllability of releases of toxic substances to the air, water, and land from manufacturing processes and subsequent product uses. This publication is one of the products of that research and provides a vital communication link between the researcher and the user community.

This study was conducted to evaluate the presence of dioxins and furans in all effluent streams from the Cincinnati Waterworks carbon reactivator and to determine the effectiveness of a recently installed afterburner in reducing or eliminating these compounds.

Francis T. Mayo, Director
Water Engineering Research Laboratory

ABSTRACT

Research was initiated to evaluate the presence of dioxins and furans in all effluent streams from the Cincinnati Waterworks carbon reactivator and to determine the effectiveness of a recently installed afterburner in reducing or eliminating these compounds. Quantitative measurements were made of (1) tetrachlorodibenzo-p-dioxins (TCDDs) and tetrachlorodibenzo-furans (TCDFs) in all effluent streams of the reactivation system and (2) penta-, hexa-, hepta-, and octa-chlorinated dioxins (CDDs) and furans (CDFs) in a preselected number of samples. The batch of carbon reactivated during this study had been used for approximately 200 days to treat an unchlorinated side stream of Cincinnati, Ohio, municipal drinking water. Seven tests were performed over a 2-week period in December 1984.

A modified Method 5 train was used to sample the stack, afterburner, and recuperator effluent streams. The train was modified by adding a water-cooled XAD-2 trap between the filter and first impinger to collect TCDDs and TCDFs in the vapor phase of the sample stream. Grab and composite sampling techniques were used to collect samples of the other effluent streams (cyclone fines, scrubber water and reactivated GAC) and the spent carbon feedstock. Natural gas and fuel oil samples used to fire the fluidized-bed reactivator were collected and analyzed for polychlorinated biphenyls (PCBs).

No 2,3,7,8-TCDD was detected in any emission and/or process streams. Tetra-CDD was not detected on any stack particulate filters, but small concentrations (<0.09 ng/dscm, or 0.006 ppt) were detected on the sorbent (XAD-2) samples from the afterburner and recuperator. Low concentrations (0.02 ng/g, or 0.001 ppt) were also found in one cyclone catch sample. Octa-CDD was detected in all emission and process streams, ranging from 3.12 ng/dscm (0.15 ppt) in recuperator particulates to 0.15 ng/g (0.007 ppt) in the spent GAC. Hepta-CDD was detected in all emission streams, ranging from 0.27 ng/dscm (0.014 ppt) in the afterburner particulates to 0.024 ng/dscm (0.001 ppt) in the vapor phase of the afterburner. Trace amounts of the hepta-CDD were also detected in one spent GAC sample. Penta- and hexa-CDD were detected in the vapor phase of the recuperator, ranging from 0.13 to 0.37 ng/dscm (0.01 to 0.02 ppt), respectively. Trace amounts of hexa-CDD were also detected in the cyclone catch.

No 2,3,7,8-TCDF was detected on the particulate filters of any emission stream, however, trace amounts (<0.049 ng/dscm) were found on the sorbent samples of all emission streams. Low concentrations (0.06 ng/g) were also observed in one cyclone catch sample. TCDF was found in recuperator emissions and in the sorbent catch of the afterburner and stack. The range was 0.01 ng/dscm (0.0007 ppt) in recuperator particulate to 1.36 ng/dscm (0.10 ppt) in the vapor phase of the recuperator. TCDF was also observed in the cyclone

catch, and in the spent GAC. Penta-CDF was detected in emissions from the afterburner and recuperator. None were detected in stack emissions. The range was from 1.07 ng/dscm (0.071 ppt) on the sorbent catch from the recuperator to 0.007 ng/dscm (0.0005 ppt) in particulate samples from the recuperator. No penta-CDF was found in any of the process samples. Hexa-CDF was detected on sorbent samples from the recuperator and afterburner, and on the filters from the afterburner and stack. The range was 0.76 ng/dscm (0.046 ppt) in the recuperator, to 0.007 ng/dscm (0.0004 ppt) in the stack. Trace amounts of the hexa-CDF were also found in the cyclone catch. Hepta-CDF was detected on sorbent samples from the recuperator and afterburner and on the filters from the afterburner and stack. The range was from 0.76 ng/dscm (0.045 ppt) on the sorbent samples from the recuperator to 0.018 ng/dscm (0.001 ppt) on the sorbent samples from the afterburner. Trace amounts (0.07 ng/g, or 0.004 ppt) of the hepta-CDF were found in the spent GAC. Octa-CDF was detected on sorbent samples from the recuperator and afterburner and on filter samples from all emission streams. The range was from 0.37 ng/dscm (0.018 ppt) on the filter samples from the recuperator to 0.012 ng/dscm (0.0006 ppt) on the sorbent samples from the afterburner. No octa-CDF was detected in any process samples.

Only one PCB isomer was detected in the natural gas supply and none in the fuel oil. The monochlorobiphenyl (Cl_1) detected in the natural gas supply was at a low level (0.71 µg/sample), which is just slightly above the detection limit of 0.18 µg/sample.

Reactivated carbon and spent carbon samples obtained during the test program and a sample of virgin carbon of the type being reactivated were analyzed for total organic halogens (TOX). The TOX levels found in the samples were: Virgin carbon, <50 mg/Kg, reactivated carbon, <50 mg/Kg, and spent carbon, 416.3 mg/Kg.

The afterburner was thus shown to have a decisive effect on reducing or eliminating dioxins and furans in the reactivator effluent streams. Post-chlorination, which was usd in this study, produced lower concentrations of these compounds than did the pre-chlorination used in an early study.

This report was submitted in fulfillment of Contract No. 68-01-6702 by Battelle-Columbus Laboratories under sponsorship of the U.S. Environmental Protection Agency. This report covers the period October 2, 1984 to June 30, 1985, and work was completed as of April 1, 1985.

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LIST OF ABBREVIATIONS

ABBREVIATIONS

kg	-- kilograms
g	-- grams
mg	-- milligrams
µg	-- micrograms
ng	-- nanograms
L	-- liters
mL	-- milliliters
µL	-- microliters
dscf	-- dry, standard cubic feet (20°C, 760mm Hg)
ft/sec	-- feet per second
cfm	-- cubic feet per minute
scf	-- standard cubic feet (20°C, 760mm Hg)
m^3	-- cubic meters
dscm	-- dry, standard cubic meters (20°C, 760mm Hg)
m/sec	-- meters per second
scm	-- standard cubic meters (20°C, 760mm Hg)
ev	-- electron volts
ms	-- milliseconds
cm	-- centimeters
amu	-- atomic mass units
µ	-- micron
ppb	-- parts per billion
ppt	-- parts per trillion

ACKNOWLEDGMENTS

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SECTION 1

INTRODUCTION

Activated carbon has been used for many years to remove taste and odor components from drinking water. More recently, studies have focused on activated carbon treatment to remove potentially hazardous trace organics from municipal water supplies. In this regard, the Cincinnati Waterworks (CWW), in conjunction with the U.S. Environmental Protection Agency (EPA) has been conducting a study of water purification employing granular activated carbon (GAC). As an economy measure, spent carbon from contactor beds is thermally reactivated in a fluidized-bed furnace and reused in the water purification process. During carbon reactivation, organic compounds could be released into effluent streams that will enter the environment. Evaluation of the net gains achieved by removing trace organics from water must include an examination of the potential for causing exposure to the public through alternative pathways. Substances removed from the water must be properly handled regardless of the treatment methods used. Thus it is important that effluent streams from the reactivation process be characterized to determine whether hazardous compounds are present.

During August and September 1982, a study was performed by Battelle's Columbus Laboratories (BCL) to measure tetrachlorodibenzo-p-dioxins (TCDD) and tetrachlorodibenzo-furans (TCDF) in the effluent streams of the CWW carbon reactivator (1). Since this work, the system has been modified by incorporation of an afterburner in the off-gas streams from the fluidized bed reactivation unit. The purpose of the afterburner is to destroy organic compounds in the off-gas stream by thermal decomposition and thus to minimize their release into the environment.

The purpose of this second study was to evaluate the effectiveness of the afterburner unit in reducing or eliminating the occurrence of dioxins and furans in the reactivator effluent streams and to determine quantitatively the TCDDs and TCDFs in all effluent streams from the Cincinnati Waterworks reactivator unit during a carbon reactivation cycle. The possible presence of TCDDs and TCDFs in the spent carbon feedstock, and PCBs in the fuel supply used to fire the reactivator were also evaluated.

The program involved collection of representative effluent streams, feedstock, and natural gas samples from the Cincinnati Waterworks fluidized-bed unit during an approximate 90-hour carbon reactivation cycle. Subsequently, laboratory analyses of the samples were performed by combined high resolution capillary column gas chromatography/high resolution mass spectrometry (HRGC/HRMS) to determine (1) 2,3,7,8-TCDD and 2,3,7,8-TCDF concentrations, (2) total TCDD and TCDF concentrations, and (3) the TCDD and TCDF isomeric

composition (i.e., the number and, when possible, the identity of isomers resolved in the analyses).

The data generated during this study along with the data obtained from the previous work will be used to evaluate any environmental and health hazards associated with effluents from reactivation of carbon used in municipal water treatment. Currently the city of Cincinnati is considering installation of a carbon purification system to treat the total municipal water supply. Thus it is important at this time to determine whether the reactivation process (an economic factor in the overall treatment costs) is environmentally acceptable.

SECTION 2

CONCLUSIONS AND RECOMMENDATIONS

Approximately 27 months elapsed between the first Cincinnati Waterworks study (1) and the second (current) study. During this period, changes were made at the municipal water purification plant and/or the GAC fluidized bed reactivation unit that could effect the removal and/or release of organic compounds into the environment. These changes included:

- Incorporation of an afterburner in the off-gas streams from the fluidized-bed reactivation unit.
- Pre-chlorination (with regard to the location of the GAC) of municipal water in the first study versus post-chlorination in the second study.
- Six months of spent GAC storage before reactivation (first study) versus 3 or 4 days in the second study.

In evaluating the effectiveness of the afterburner in reducing or eliminating the occurrence of dioxins and furans, we made some comparisons of stack emissions in the first and second studies. In the first study, TCDDs were detected in stack emission samples from all four tests. In the second study, TCDDs were not detected in stack emissions in two tests. Also in the first study, TCDFs were detected in stack emission samples in three of four tests. In the second study, TCDFs were detected in only one stack emission sample. Since analyses of the higher homologues of dioxins and furans were only obtained in the second study, no comparison of the higher homologues can be made with the first study. Conclusions drawn from comparable data from the first and second studies indicate that the afterburner has a decisive effect in reducing or eliminating the occurrence of dioxins and furans in the reactivator effluent streams.

The location of GAC in the water purification scheme with regard to chlorination appears to be important. Pre-chlorination (employed in the first study) may increase the availability of chlorine precursors that form by-products and, when heated, increase the probability of additional dioxin and/or furan formation. This hypothesis was borne out in the second study (post-chlorination), when lower concentrations of TCDDs and TCDFs were observed.

The data generated during this study provide a reasonable data base for evaluating the environmental impact of the municipal fluidized-bed GAC reactivation system.

The study showed no evidence (pro or con) of any side effects evolving from the storage of spent GAC (up to 6 months) before reactivation. Additional work currently being performed on the carbon reactivation system at the Jefferson Parish, Louisiana, Department of Water should provide more information on the overall environmental acceptability of the GAC reactivation process.

SECTION 3

EXPERIMENTAL PROCEDURES

GENERAL EXPERIMENTAL APPROACH

Seven tests were performed to determine TCDD and TCDF emissions from the Cincinnati Waterworks carbon reactivation unit. The test sequence and sampling matrix is shown in Table 1. The table shows the test sequence and for each test, the system operating variables, the samples that were analyzed, and the type of analysis that was performed. The tests were conducted during reactivation of a batch of carbon which had been used to adsorb trace organics from Cincinnati municipal drinking water. During testing, samples were acquired from all effluent streams and the spent carbon feedstock. Fuel samples were collected and analyzed for polychlorinated biphenyls (PCBs). The process streams that were sampled included:

(1) Stack effluent (particulate and sorbent catches) from the fluidized-bed unit after passing through the Venturi and tray scrubbers

(2) Afterburner effluent (1 and 2 second residence time)

(3) Spent GAC feed

(4) Reactivated GAC

(5) Dryer off-gas cyclone fines

(6) Venturi/tray scrubber water

(7) Heat exchanger inlet

(8) Reactivator fuel feed.

A modified Method 5 train was used to sample the afterburner, recuperator and stack effluent streams. Grab and composite sampling techniques were used to collect samples of the other effluent streams and the spent carbon feedstock. During two of the tests, the natural gas and fuel oil supply used to fire the fluidized-bed reactivator were sampled with a solid sorbent trap (XAD-2) to screen for the presence of PCBs.

The effluent streams and spent carbon feed samples were analyzed by extraction, removal of co-extracted interferences, and determination of TCDDs and TCDFs by combined capillary column gas chromatography/high resolution

TABLE 1. TEST SEQUENCE AND SAMPLING MATRIX FOR CINCINNATI WATERWORKS CARBON REACTIVATOR TESTS

Test Sequence	Reactivator Fuel[a]	Afterburner Off/On	GAC Feed	Process Streams to be Sampled[b]								
				Spent GAC	Regen. GAC	Scrubber Water	Carbon Fines	AB[c] 1 sec	AB[c] 2 sec	Recup. Inlet	Stack[c]	Fuel Supply
1	NG	Off	No			X				TP		P
2	NG	Off	Yes	X	X	X	T			T		
3	NG	On	Yes	X	X	T	X		T		T	X
4	NG	On	Yes	X	X	X	X	T				
5	NG	On	Yes	TP	TP	TP	X		TP		TP	X
6	NG	Off	Yes	X	X	X	TP			TP		X
7	FO	Off	No			X				T		P
No. Samples To be Analyzed				1	1	2	2	2	4	8	4	2

a) NG - Natural gas, FO - Fuel oil.
b) X - Sample collected and archived.
T - Sample collected and analyzed for TCDD/TCDF only.
TP - Sample collected and analyzed for TCDD/TCDF and PCDD/PCDF.
P - Sample collected and analyzed for PCB.
c) Modified Method 5 train with a sorbent (XAD-2) used for each test.

mass spectrometry. Analyses of the sorbent traps used to sample the fuel supply were done by extraction and determination of the PCBs by combined capillary column gas chromatography/mass spectrometry.

The following sections provide more detailed descriptions of the carbon reactivation process and the sampling and analytical procedures that were employed in the study.

PROCESS DESCRIPTION AND CONDITIONS

The purpose of the reactivation process is to restore the absorptive properties of GAC so that it may be reused for water purification. A schematic diagram of the Cincinnati Waterworks fluidized-bed carbon reactivation system is presented in Figure 1. The system functions consist of drying the spent carbon feed and thermally-reactivating the GAC in a fluidized bed. The off-gases from the unit are passed through a recently-installed afterburner and treated by wet scrubbing prior to release to the atmosphere.

The wet spent carbon is fed into the reactivation unit by a dewatering screw and first passes through a drying zone which is maintained at about 149°C (300°F). The carbon then passes into a fluidized bed reactivation zone in which the temperature is closely controlled at about 816°C (1500°F) to minimize carbon burnup losses. The unit may be fired with either natural gas or fuel oil with the former fuel being used in the normal operating mode.

Off gases from the dryer and reactivation zone pass through the afterburner/heat recovery unit, then through serially-connected Venturi and tray scrubbers, and are emitted to the atmosphere through a stack. Particulate material collected in the dryer off gas cyclone is fed through a grinder and then injected into the afterburner unit. The reactivated GAC drops into a water quench tank and is recovered as a slurry for reuse in water treatment.

Reactivator operating data were recorded and archived by Cincinnati Waterworks personnel during the test program. A summary of operating conditions during the test periods is given in Table 2. Stack gas parameters determined by EPA Method 5 procedures are shown in Table 3.

The emission tests were performed during reactivation of about a 18,144 Kg (40,000 lb) batch of WestVaco bituminous carbon (12 x 40 mesh) which had been used for treatment of Cincinnati municipal water. The carbon was in service in a contactor bed treatment system for approximately 200 days. About four days elapsed from the time the carbon was taken out of service and the reactivation was performed.

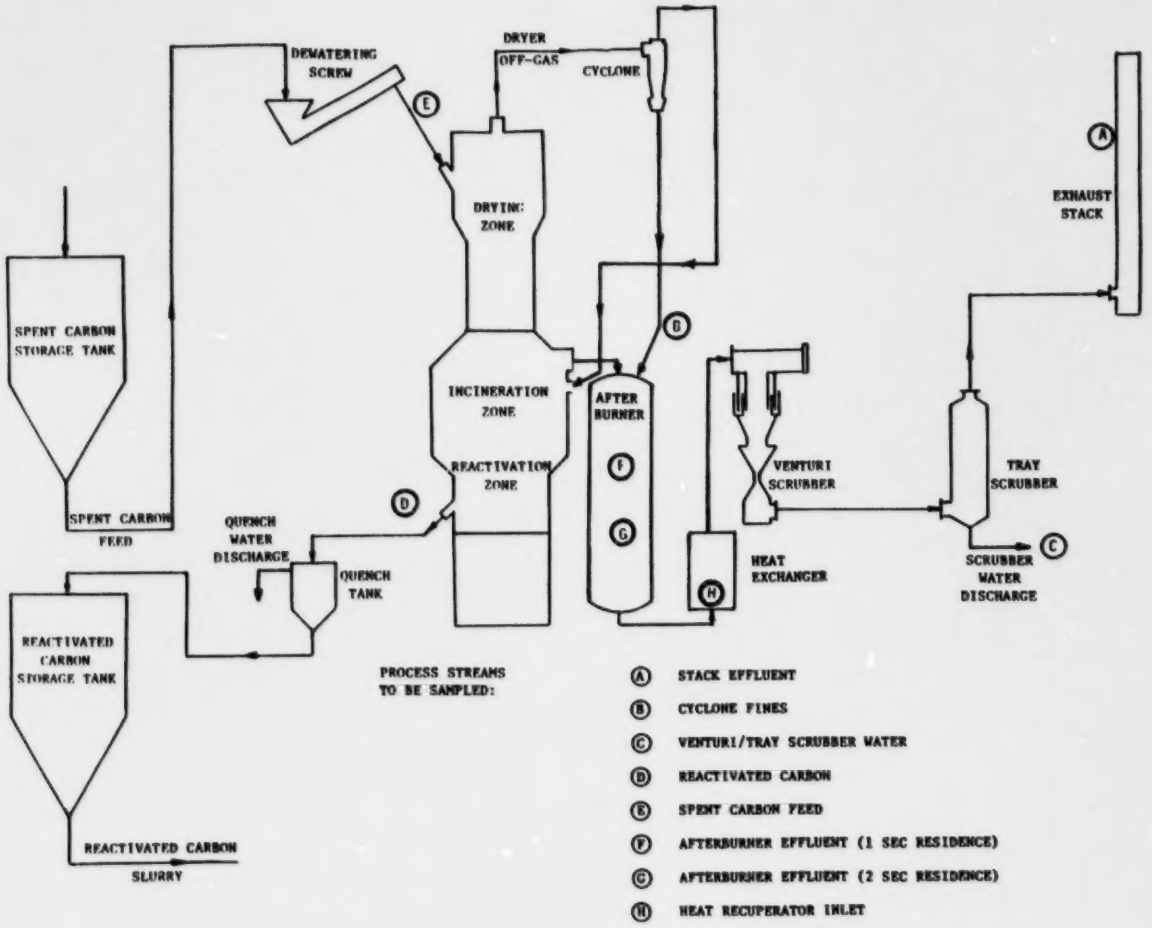


Figure 1. Cincinnati Waterworks carbon reactivation system.

TABLE 2. SUMMARY OF OPERATING CONDITIONS DURING EMISSION TESTS[a]

	1-Recup.	2-Recup.	3-AB, 2 sec.	3-Stack	4-AB, 1 sec.	5-AB, 2 sec.	5-Stack	6-Recup.	7-Recup.
Spent Carbon Feed[b]									
Rate, Kg/hr(lbs/hr)[c]	Off	265.4(585)	265.4(585)	265.4(585)	265.4(585)	265.4(585)	265.4(585)	265.4(585)	Off
Total, Kg(lbs)	Off	1326.8(2925)	1326.8(2925)	1326.8(2925)	1326.8(2633)	1326.8(2925)	1326.8(2925)	1459.7(3218)	Off
Nat. Gas Feed[d]									
Rate, $m^3/m(ft^3/m)$	0.9(31.9)	1.0(36.0)	0.8(29.3)	0.8(29.3)	0.9(30.5)	0.9(31.8)	0.9(31.8)	0.9(31.4)	0.2(6.7) 19.5[e]
Total, $m^3(ft^3)$	262.3(9270)	304.8(10770)	323.4(11427)	323.4(11427)	311.0(10990)	269.8(9534)	269.8(9534)	293.0(10353)	56.9(2010) 92[e]
Comb. Chamber									
Temp., °C (°F)	685(1266)	946(1737)	796(1466)	796(1466)	768(1415)	908(1667)	908(1667)	977(1790)	948(1738)
O_2, %	0	0	0.7	0.7	0.4	0.3	0.3	1.0	2.7
Reactivator Bed									
Temp., °C (°F)	842(1547)	872(1603)	855(1571)	855(1571)	821(1511)	852(1566)	852(1566)	830(1526)	848(1558)
Incinerator									
Temp., °C (°F)	848(1558)	982(1798)	921(1688)	921(1688)	896(1644)	904(1660)	904(1660)	935(1716)	793(1459)
O_2, %	3.1	2.6	1.6	1.6	2.2	1.7	1.7	2.5	3.0
Afterburner									
Temp., °C (°F)	700(1294)	845(1554)	1350(2463)	1350(2463)	1333(2432)	1344(2453)	1344(2453)	749(1372)	549(1020)
O_2, %	5.0	4.5	2.1	2.1	2.1	2.3	2.3	3.5	9.6
Dryer Bed Temp., °C (°F)	154(309)	153(308)	158(317)	158(317)	153(307)	148(298)	148(298)	157(315)	162(324)
Scrubber Gas Temp., °C (°F)									
Vent. Out	59(138)	67(153)	80(176)	80(176)	79(175)	79(174)	79(174)	62(143)	32(90)
Tray Out	34(93)	37(98)	51(124)	51(124)	54(129)	52(125)	52(125)	25(77)	22(71)

(a) Data are totals or averages for the approximate time periods during which the emission tests were performed.
(b) Multiply by 0.4536 to convert lbs or lbs/hr to Kg or Kg/hr.
(c) Designed rate.
(d) Multiply by 0.02832 to convert cf or cfm to m^3 or m^3/min.
(e) Fuel oil fired in reactivator, 19.5 gal/hr and 92 total gallons.

TABLE 3. SUMMARY OF STACK GAS PARAMETERS

Parameters	Test No. 1 Recup.	Test No. 2 Recup.	Test No. 3 AB2	Test No. 3 STK	Test No. 4 AB1	Test No. 5 AB2	Test No. 5 STK	Test No. 6 Recup.	Test No. 7 Recup.
CO_2 in Stack Gas, % by vol.	8.0	8.9	-	12.75	-	-	11.9	10.5	6.25
O_2 in Stack Gas, % by vol.	12.0	12.6	-	5.0	-	-	5.4	13.0	15.3
H_2O in Stack Gas, % by vol.	41.4	33.1	30.3	7.7	32.0	31.2	8.9	30.0	28.5
Avg. Stack Gas Temp., °C (°F)	681.0 (1,257.0)	860.0 (1,579.0)	1,337.0 (2,441.0)	39.0 (102.5)	1,334.8 (2,435.0)	1,329.0 (2,424.0)	41.1 (106.0)	766.5 (1,413.0)	493.3 (920.0)
Stack Gas Velocity, ft/sec	35.30	46.37	5.40[a]	81.49	5.40[a]	5.40[a]	81.85	56.39	29.78
Stack Gas Velocity, m/sec	10.76	14.13	1.64[a]	24.84	1.64[a]	1.64[a]	24.95	17.19	9.08
Volumetric Flow Rate, dscf/hr	50,188.13	63,164.69	81,948.53	99,265.33	67,846.74	68,300.79	97,874.19	87,199.61	63,676.74
Volumetric Flow Rate, dscm/hr	1,421.18	1,787.56	2,319.14	2,809.21	1,909.87	1,932.91	2,769.84	2,467.75	1,802.05

(a) Estimated from operational data.

SAMPLING PROCEDURES

Stack, Afterburner and Heat Exchanger Effluent

A modified Method 5 train, shown schematically in Figure 2, was used to sample the stack, afterburner and heat exchanger effluent. The train is similar to a system which has been used to assess organic emissions from combustion sources (2). The modification to the conventional Method 5 train consisted of the addition of a water-jacketed condenser and XAD-2 trap between the filter and the first impinger. Cooling water to the condenser and XAD-2 trap jacket was supplied by a submersible pump located in the impinger ice bath. Liquid which condensed and passed through the XAD-2 resin bed was collected in a reservoir and retained for TCDD/TCDF analyses.

Stack emission tests were performed by sampling isokinetically at a single point in the stack in a region of average velocity (ΔP) as determined from the preliminary traverse data shown in Appendix A. A Pyrex-lined, heated probe was used to extract the stack sample and maintain the gas at the temperature specified in EPA Method 5.

The afterburner effluent tests were performed at ports in the afterburner located at positions corresponding to one and two second gas residence times. Two ports at 90^{o} angles are located at each residence time position. The tests were conducted by sampling isokinetically for a five minute period at each of 24 points on traverses at each port. Thus, 48 points in the afterburner effluent were sampled during each test.

The modified Method 5 procedure was also used to obtain reactivator effluent stream samples at the recuperator inlet ports. Two ports, at 90^{o} angles, were sampled isokinetically for a 15-minute period at each of eight points on traverses at each port. A total of 16 points at the recuperator inlet were sampled during each test. As in the afterburner tests, a water-cooled stainless steel/Incoloy probe was used to cool the sample-gas stream to the temperature specified by EPA Method 5.

Carbon dioxide (CO_2) and oxygen (O_2) concentrations in the stack, afterburner and heat exchanger, were obtained at least four times during each Method 5 test using Fyrite analyzers. Results of the CO_2 and O_2 measurements and sampling system operating data recorded during Test No. 3 are given in Appendix A. A summary giving test dates and times and the modified Method 5 operating conditions for each test is presented in Table 4.

Following the modified Method 5 tests, the components of the sampling train were sealed and moved to a clean laboratory area at the Cincinnati Waterworks to conduct sample recovery operations. Recovery of the various samples from the sampling system was performed as shown in Figure 3. Sample recovery data for Test No. 3 are presented in Appendix A.

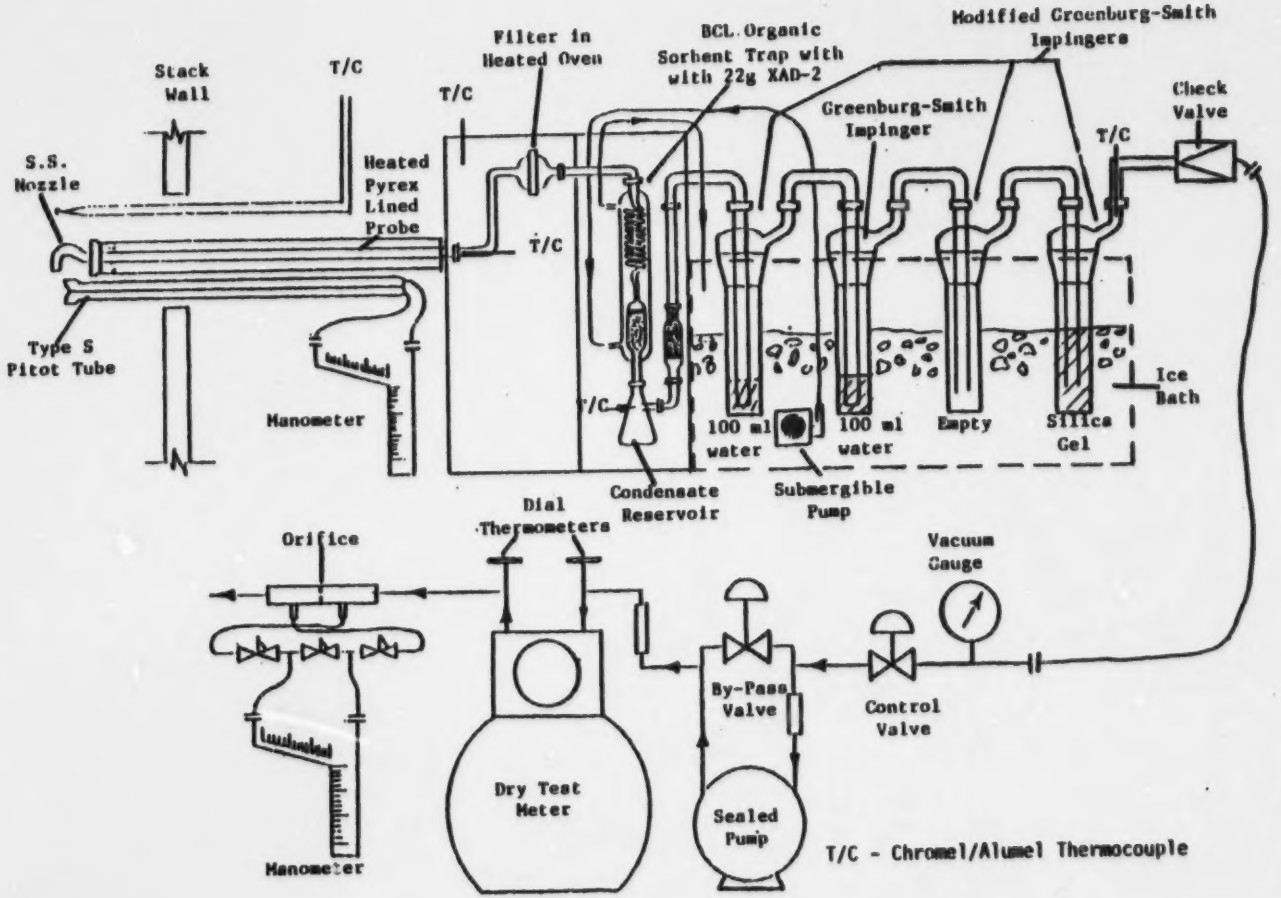


Figure 2. Modified method 5 sampling train.

TABLE 4. SUMMARY OF MODIFIED METHOD 5 SYSTEM OPERATING CONDITIONS

Parameter	Test No. 1 Recup.	Test No. 2 Recup.	Test No. 3 AB2	Test No. 3 STK	Test No. 4 AB1	Test No. 5 AB2	Test No. 5 STK	Test No. 6 Recup.	Test No. 7 Recup.
Test Date	12/4/84	12/5/84	12/8/84	12/8/84	12/11/84	12/12/84	12/12/84	12/13/84	12/14/84
Time Started, hr EDT	1302	1203	1416	1417	1130	0050	0048	1139	0930
Time Completed, hr ETD	1802	1709	1930	1930	1550	0600	0600	1700	1415
Sampling Time, min.	240	240	240	240	240	240	240	240	240
Sample Volume, dscf	135.660	159.094	110.418	127.198	108.244	111.247	125.781	182.081	132.833
Sample Volume, dscm	3.842	4.506	3.127	3.602	3.065	3.151	3.562	5.157	3.762
Isokinetic Sampling Rate, %	107.9	100.5	109.5	95.9	105.3	106.9	96.2	84.0	83.3
Avg. Probe Temp., °C (°F)	149 (300)	190 (374)	117 (242)	126 (259)	94 (202)	98 (209)	125 (257)	111 (232)	117 (242)
Avg. Filter Box Temp., °C (°F)	121 (250)	122 (251)	127 (260)	113 (236)	138 (281)	144 (292)	118 (244)	129 (264)	127 (261)
Avg. XAD-2 Outlet Temp., °C (°F)	11 (52)	9 (48)	7 (44)	11 (51)	14 (58)	5 (41)	9 (48)	7 (45)	4 (40)

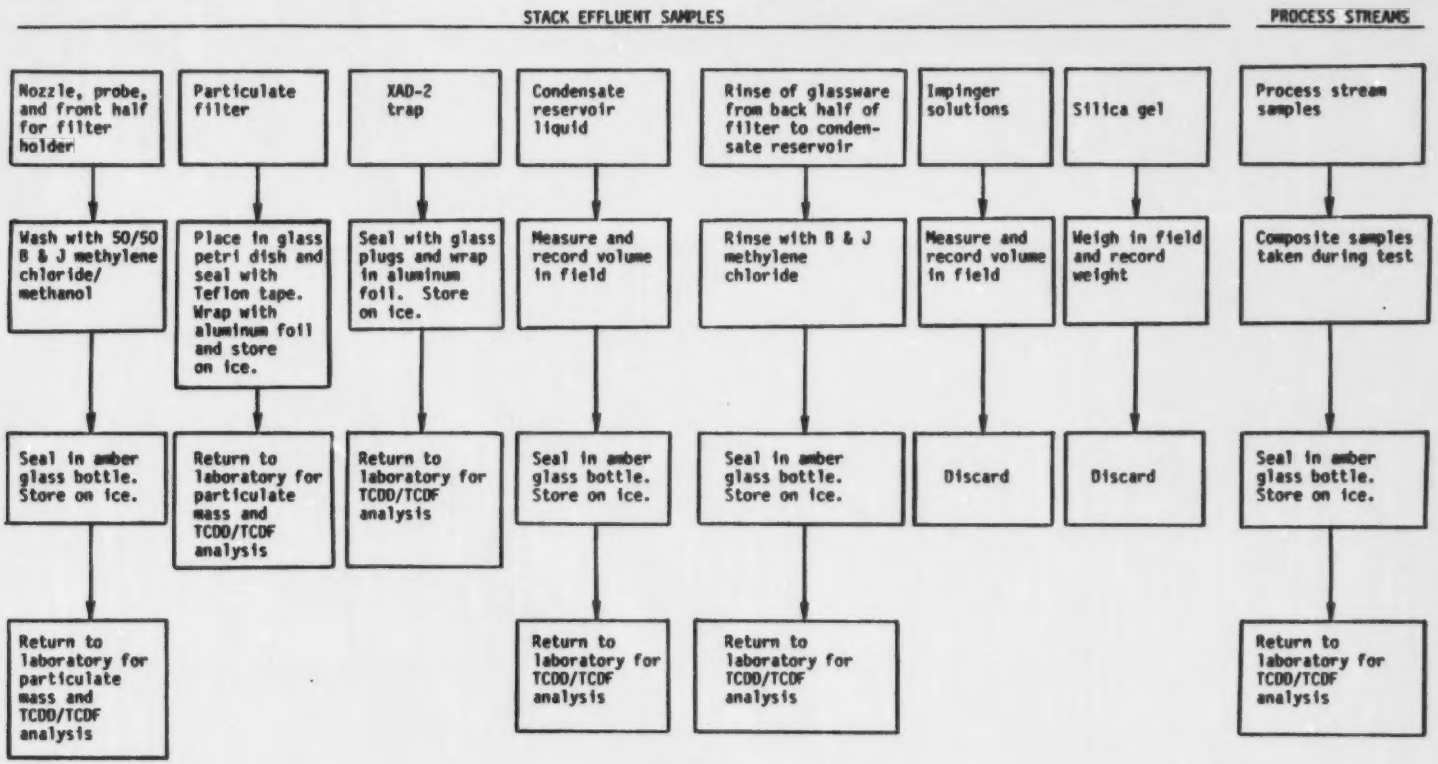

STACK EFFLUENT SAMPLES
PROCESS STREAMS
Nozzle, probe, and front half for filter holder
Particulate filter
XAD-2 trap
Condensate reservoir liquid
Rinse of glassware from back half of filter to condensate reservoir
Impinger solutions
Silica gel
Process stream samples
Wash with 50/50 B & J methylene chloride/ methanol
Place in glass petri dish and seal with Teflon tape. Wrap with aluminum foil and store on ice.
Seal with glass plugs and wrap in aluminum foil. Store on ice.
Measure and record volume in field
Rinse with B & J methylene chloride
Measure and record volume in field
Weigh in field and record weight
Composite samples taken during test
Seal in amber glass bottle. Store on ice.
Return to laboratory for particulate mass and TCDD/TCDF analysis
Return to laboratory for TCDD/TCDF analysis
Seal in amber glass bottle. Store on ice.
Seal in amber glass bottle. Store on ice.
Discard
Discard
Seal in amber glass bottle. Store on ice.
Return to laboratory for particulate mass and TCDD/TCDF analysis
Return to laboratory for TCDD/TCDF analysis
Return to laboratory for TCDD/TCDF analysis
Return to laboratory for TCDD/TCDF analysis

Figure 3. Field sample recovery procedures.

Spent Carbon, Reactivated Carbon, and Cyclone Fines

Approximately 200 ml grab samples of the spent carbon, reactivated carbon, and cyclone fines were taken at 30-minute intervals during modified Method 5 tests. The eight grab samples taken from each stream during a test were combined and homogenized to provide a single composite sample of each type for analysis. The cyclone fines sample was taken at the discharge of the cyclone and before grinding. The spent carbon feed sample was acquired at a port in the drop line from the dewatering screw to the dryer. Samples of reactivated carbon were obtained from a double-valved sampling port in the drop chute to the quench tank. Field recovery procedure for the spent carbon, reactivated carbon, and cyclone catch samples is shown in Figure 3.

Scrubber Water

Sixteen-ounce grab samples of the scrubber water were taken at 30-minute intervals during each modified Method 5 test. The eight grab samples taken during the test were combined and homogenized by shaking to yield a single composite sample as shown in Figure 3. The grab samples were obtained from a tap in the scrubber water effluent line. The tap line was thoroughly flushed just prior to sample collection, and the 16-oz collection bottles were rinsed three times with the scrubber water prior to acquisition of the sample.

Reactivator Fuel Supply

During Test No. 1 and 7, the natural gas and the fuel oil supply used to fire the fluidized-bed reactivation system were sampled to determine if polychlorinated biphenyls (PCBs) were present.

The natural gas supply was sampled by passing a sample stream from the supply line to the boiler through a 22-gram XAD-2 trap operated at ambient temperature. Sample volume was measured with a calibrated dry gas meter downstream from the XAD-2 trap. Sampling was performed for a two hour period during the four hour modified Method 5 test. Flow rate through the XAD-2 trap was regulated at approximately 0.014 m^3/min (0.5 cfm), with a total sample volume of 1.77 m^3 (62.7 cf).

During operation of the reactivation unit on fuel oil, 16 oz. (500 ml) grab samples of the fuel feed were taken hourly during the four hour modified Method 5 test. The four samples were composited to provide a single sample for PCB analysis.

Preparation of Sampling Equipment and Sample Containers

All glassware used for sampling and sample containment was cleaned prior to use by the following steps:

(1) Wash in a hot synthetic detergent solution

(2) Rinse with hot tap water

(3) Soak for at least 30 minutes in hot 40-50°C (104-122°F) chromic acid solution*

(4) Rinse with hot tap water

(5) Rinse with distilled water

(6) Rinse with methanol (Burdick & Jackson "Distilled in Glass")

(7) Rinse with methylene chloride (Burdick & Jackson "Distilled in Glass")

(8) Dry with a stream of purified nitrogen.

After drying, all equipment and containers were sealed to prevent contamination prior to sampling.

Sample Containment and Preservation

Following collection, all samples were transferred to amber glass bottles with Teflon cap liners or clear glass containers wrapped with aluminum foil. The samples were then placed on ice (0°C or 32°F) in ice chests for storage during the sample collection period and during shipment to Battelle's laboratories. Upon arrival at the laboratory, all samples were transferred from the ice chests to refrigerated storage where they were held until start of the analysis. Analysis of the samples commenced 6 days after the samples were delivered to the laboratory.

ANALYTICAL PROCEDURES

TCDD and TCDF Analyses

Overview of Sample Analysis Steps--

TCDD and TCDF analyses of the samples involved three basic steps: (1) extraction of the TCDDs and TCDFs from the sample matrix; (2) sample clean-up to remove co-extracted interferences; and (3) analysis of TCDDs and TCDFs by HRGC/HRMS. Several intermediate steps were required to reduce sample volume and to exchange the extract to a solvent suitable for subsequent operations. Figures 4 and 5 show the sequence of the steps in the TCDD/TCDF analysis procedure. Figure 4 shows the sample preparation/ combination and extraction steps for each sample type. The cleanup and analysis steps which were common to all sample types are shown in Figure 5.

* 15% HNO_3 was used in this step to clean the modified Method 5 stainless steel sampling nozzle.

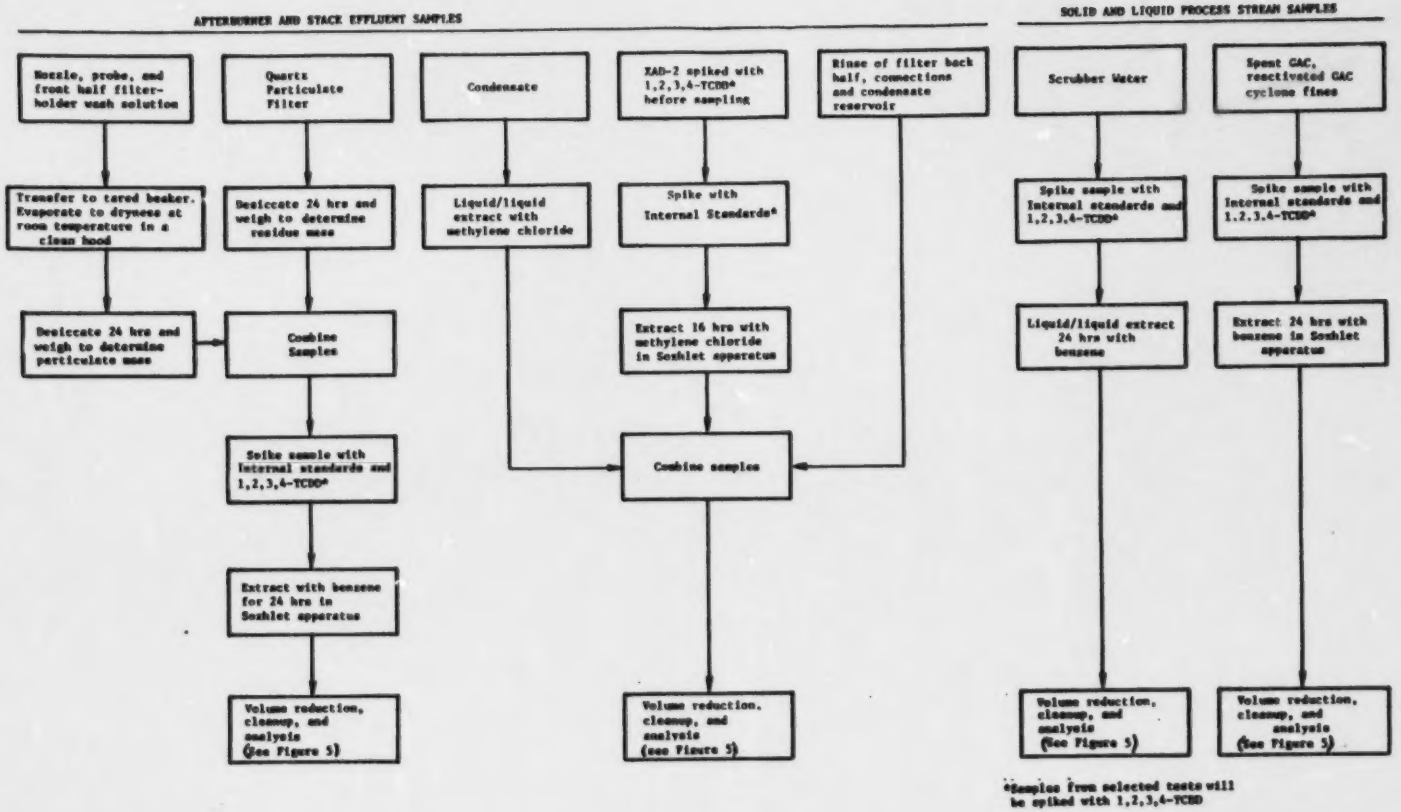

APTERBURNER AND STACK EFFLUENT SAMPLES
SOLID AND LIQUID PROCESS STREAM SAMPLES
Nozzle, probe, and front half filter-holder wash solution
Quartz Particulate Filter
Condensate
XAD-2 spiked with 1,2,3,4-TCDD* before sampling
Rinse of filter back half, connections and condensate reservoir
Scrubber Water
Spent GAC, reactivated GAC cyclone fines
Transfer to tared beaker. Evaporate to dryness at room temperature in a clean hood
Desiccate 24 hrs and weigh to determine residue mass
Liquid/liquid extract with methylene chloride
Spike with Internal Standards*
Spike sample with Internal standards and 1,2,3,4-TCDD*
Spike sample with Internal standards and 1.2.3.4-TCDD*
Desiccate 24 hrs and weigh to determine particulate mass
Combine Samples
Extract 16 hrs with methylene chloride in Soxhlet apparatus
Liquid/liquid extract 24 hrs with benzene
Extract 24 hrs with benzene in Soxhlet apparatus
Spike sample with Internal standards and 1,2,3,4-TCDD*
Combine samples
Extract with benzene for 24 hrs in Soxhlet apparatus
Volume reduction, cleanup, and analysis (See Figure 5)
Volume reduction, cleanup, and analysis (see Figure 5)
Volume reduction, cleanup, and analysis (See Figure 5)
Volume reduction, cleanup, and analysis (See Figure 5)
*Samples from selected tests will be spiked with 1,2,3,4-TCDD

Figure 4. Sample combination/preparation steps.

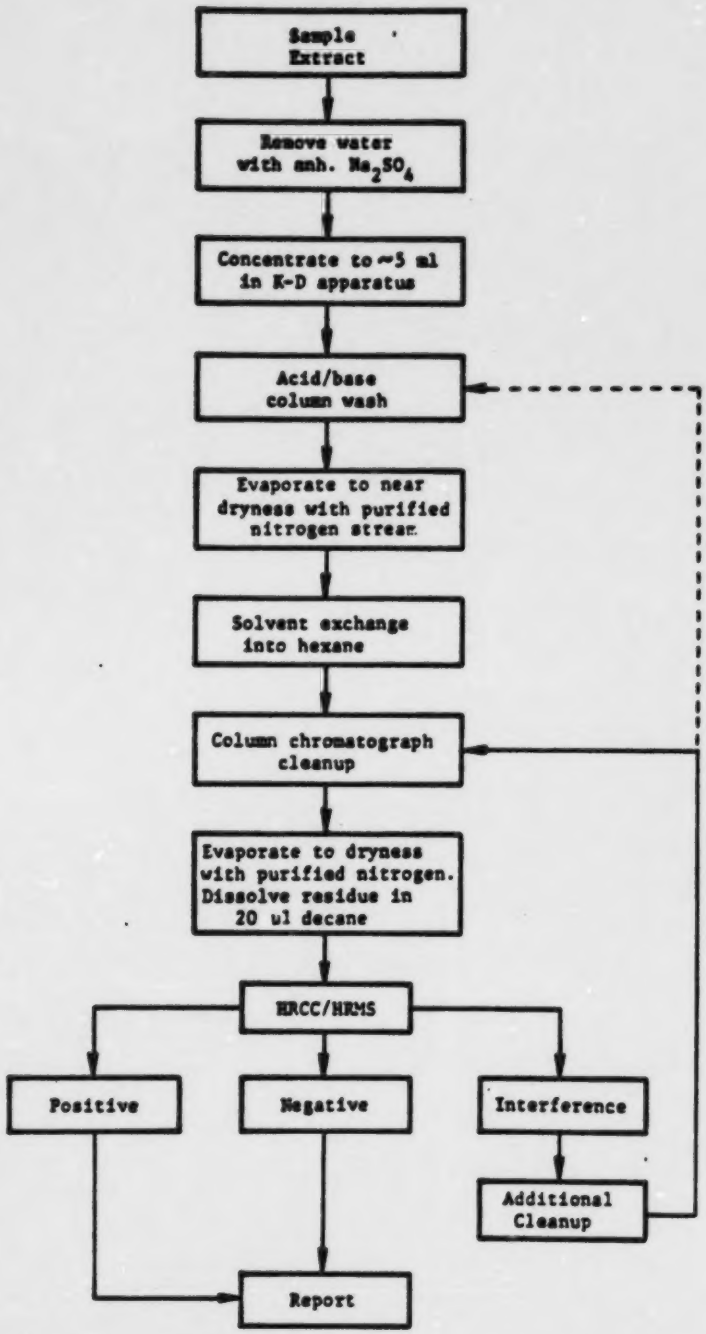


Figure 5. Sample clean-up and analysis steps.

Extraction Procedures--

Two extraction techniques were used for the sample analyses. The XAD-2 resin/organic module rinse samples and the particulate samples were Soxhlet-extracted with methylene chloride and benzene, respectively, while liquid-liquid extraction was used for the scrubber and quench water samples. Methylene chloride is commonly used as the solvent for most XAD-2 Soxhlet extractions, and has been shown to be an efficient solvent for the extraction of TCDD/TCDF. The particulate samples however, due to stronger adsorption, require an aromatic solvent such as benzene to assure efficient recovery of the TCDDs and TCDFs (3). Prior to extraction, each sample was spiked with 2,3,7,8-TCDD-$^{13}C_{12}$ and 2,3,7,8-TCDF$^{13}C_{12}$ to serve as internal standards for recovery calculations and quantification. A description of the extraction methodologies follows.

The particulate samples included the combined modified Method 5 filter and rinse residue, the dryer off-gas cyclone catch, and spent carbon feed samples.

For the extraction, the particulate samples were transferred into the extraction thimble and placed into the Soxhlet apparatus. The 2,3,7,8-TCDD-$^{13}C_{12}$ (20 ng) internal standard was added and the samples were extracted with benzene. Teflon tape was used to seal the ground glass joints to minimize evaporative losses. Following the extraction, the benzene was transferred to an Erlenmeyer flask using three 15 ml rinses of fresh benzene. The benzene was dried by slowly adding approximately 1 g of anhydrous sodium sulfate. The sodium sulfate was added slowly to prevent the formation of clumps which can entrap the TCDDs and TCDFs. The dried extract was then transferred to a Kuderna-Danish (K-D) apparatus using three 15 ml rinses of fresh benzene and concentrated to approximately 5 ml volume. The extract was stored in a glass stoppered bottle at 0°C(32°F) until processed through the acid/base and chromatographic clean-up steps.

The XAD-2 resin samples were Soxhlet-extracted using methylene chloride. The resin was transferred into a pre-extracted cellulose extraction thimble, spiked with 2 ng of 2,3,7,8-TCDD-$^{13}C_{12}$ and 2 ng of 2,3,7,8-TCDF-$^{13}C_{12}$ and allowed to extract for at least 16 hours. The methylene chloride extract was dried with anhydrous sodium sulfate and concentrated in a K-D apparatus to a volume of approximately 5 ml following the same procedure as described for the particulate extracts.

The scrubber water samples were extracted by liquid-liquid extraction using benzene. A one-liter aliquot of each sample was transferred to a two-liter glass bottle and spiked with 20 ng of 2,3,7,8-TCDD-$^{13}C_{12}$ and 20 ng of 2,3,7,8-TCDF-$^{13}C_{12}$. A Teflon-coated magnetic stirring bar was added and the sample was stirred for about 30 minutes. A 300 ml portion of benzene was added and the stirring resumed for at least 24 hours. The benzene extract was then transferred to an Erlenmeyer flask using three 50 ml rinses of fresh benzene, dried with anhydrous sodium sulfate, and concentrated to 5 ml using the procedure described for the particulate extracts.

Sample Clean-Up Procedures--

The benzene extracts of the modified Method 5 particulate, scrubber water, and spent carbon feed samples and the methylene chloride extracts of the condensate and XAD-2 resin (including the methylene chloride organic module rinse) were treated to remove interferences using a series of liquid chromatography (LC) columns. The first column used to treat the extracts was a multilayered silica column which served to remove acidic, basic, and easily oxidized compounds. The column contained approximately 4 g of silica treated with sulfuric acid, 4 g of silica treated with sodium hydroxide, and 2 g of activated silica. The column was packed dry and pre-rinsed with 25 ml of hexane prior to use.

The sample extracts were transferred onto the silica column using three 5 ml rinses of hexane/benzene (50:50). The extract was eluted with 50 ml of hexane/benzene (50:50) and the total eluate (about 70 ml) was collected. The eluate was then concentrated nearly to dryness under a gentle stream of purified nitrogen and dissolved in about 5 ml of hexane.

The extract (in hexane) was transferred to a 5 g activated alumina clean-up column. The column was pre-rinsed with 25 ml hexane followed by the addition of the sample. The column was sequentially eluted with 25 ml portions of hexane, hexane/carbon tetrachloride (1:1), and hexane/methylene chloride (1:1). The hexane/methylene chloride fraction was collected and stored at 10°C (50°F) until analyzed. Just prior to analysis, the hexane/methylene chloride solution was evaporated to dryness at ambient temperature using a stream of purified nitrogen. The residue was then dissolved in 20 µl of n-decane (accurately measured). Analysis for TCDD and TCDF was performed on a 2 µl aliquot of the n-decane solution.

HRGC/HRMS Analysis of TCCDs and TCDFs--

The extracts were analyzed for TCDDs and TDCFs using combined capillary column gas chromatography/high resolution mass spectrometry (HRGC/HRMS). A Carlo Erba Model 4160 gas chromatograph interfaced to a VG Model 7070H high resolution mass spectrometer was used for the analyses. Typical operating parameters were:

Mass Spectrometer		
Electron Impact Ionization	-	70 ev
Mass Resolution	-	10,000-12,000 (m/ m, 10% valley)
Source Temperature	-	200°C (392°F)
Multiple Ion Detection Mode	-	25 ms dwell time, total cycle time 200 ms
Gas Chromatograph		
Column	-	30 meter DB5 or 50 meter CPS:1-88 Fused Silica Column
Carrier Flow	-	30 cm/sec (helium)
Injector Temperature	-	275°C (527°F)

Temperature Program	-	140°C (284°F) for 3 minutes; linear increase to 275°C (527°F) at 20°C (36°F)/ minute and hold at 275°C (527°F) for 10 minutes
Transfer Line	-	290°C (554°F)

A VG Model 2035 Data System was used to control the multiple ion detection (MID) acquisition and to acquire the time-intensity data. Seven ion masses shown below were monitored.

318.9793	Reference Lock Mass (Perfluorokerosene)
303.9016	TCDF
305.8987	TCDF
319.8965	TCDD
321.8936	TCDD
331.9368	2,3,7,8-TCDD-$^{13}C_{12}$ internal standard
333.9338	2,3,7,8-TCDD-$^{13}C_{12}$ internal standard

The criteria for a positive identification were:

1. Correct chromatographic retention time within ±2 seconds based on 2,3,7,8-TCDD-$^{13}C_{12}$ internal standard and external standards (1,3,6,8-TCDD, 1,3,7,9-TCDD, 2,3,7,8-TCDD, or 2,3,7,8-TCDF)

2. Response at both m/z 304 and m/z 306 for TCDFs

3. Response at both m/z 320 and m/z 322 for TCDDs

4. Isotopic ratio correct for a tetrachloro compound (0.77 ± 15%)

5. Signal-to-noise ratio equal to or greater than 2.5 to 1.0.

The TCDDs and TCDFs were quantified by comparison of the responses (selected ion current profile peak areas), of the native TCDD/TCDF isomers with the response of the 2,3,7,8-TCDD-$^{13}C_{12}$ internal standard. Thus all reported TCDD/TCDF levels were corrected for the recovery efficiencies. A response factor was calculated from triplicate analyses of standard solutions.

PCB Analysis--

Analysis for PCBs collected from the natural gas supply line by trapping on XAD-2 was performed by solvent extraction of the solid sorbent and determination of PCBs in the extract by combined high resolution gas chromatography/mass spectrometry (HRGC/MS). The XAD-2 was extracted with methylene chloride for about 16 hours in a Soxhlet apparatus. The extract was then reduced in volume to 0.2 mL in a Kuderna-Danish apparatus followed by evaporation with a stream of purified nitrogen. The extract was next spiked with 2 µg of dibromobiphenyl (internal standard) and analyzed by HRGC/MS using a

Finnigan Model 3200 system with an INCOS data system. The HRGC/MS operating conditions were as follows:

Mass Spectrometer
- Electron Impact Ionization - 70 ev
- Source Temperature - 100°C (212°F)
- Scan Range - 40 to 540 amu
- Scan Rate - 0.75 sec/scan
- Mode - Full spectrum scan

Gas Chromatograph
- Column - 30 meter, DB-5 Fused Silica
  0.248 in. ID, 0.25μ film (J&W Scientific)
- Carrier flow - 30 cm/sec
- Temperature program - 45°C (113°F) for 4.5 minutes; linear increase to 300°C (572°F) at 8°C (14°F)/minute
- Injector temperature - 300°C (572°F)
- Transfer Line Temperature - 300°C (572°F)
- Injector - Grob-type, splitless
- Injection volume - 2 μl

The PCB concentration in the XAD-2 extract was determined by comparison to a standard solution comprised of known quantitites of the PCB isomers listed in Table 5. The response factor for each level of chlorination was determined by summation of the ion currents for the two characteristic masses specific to that chlorination level (see Table 5). The response factors obtained from the standard solution were applied to the sample extract to determine the concentration of all isomers at each level of chlorination. The response for all isomers of a given chlorination level in the sample was assumed to be the same as the response for the isomer used as the reference standard.

Each selected ion current profile for a given level of chlorination was manually examined to assure that both quantitation masses were present and that they maximized at the same scan number. The isotope ratio for the two masses was also considered.

TABLE 5. COMPOSITION OF PCB STANDARD[a]

Level of Chlorination	Isomer	Masses used for Quantitation (m/z)
1	2-chlorobiphenyl	188,190
2	2,6-dichlorobiphenyl	222,224
3	2,2'5-trichlorobiphenyl	256,258
4	2,2'5,6'-tetrachlorobiphenyl	290,292
5	2,2',3,5,6-pentachlorobiphenyl	326,328
6	2,2',4,4',5,6-hexachlorobiphenyl	360,362
7	2,2',3,4,4',5,6-heptachlorobiphenyl	394,396
8	2,2'3,3'4,5,6,6'-octachlorobiphenyl	428,430
Internal Standard	4,4'-dibromobiphenyl	310,312

(a) Standard solution prepared from individual PCB isomers obtained from Ultra Scientific.

SECTION 4

RESULTS AND DISCUSSION

STACK PARTICULATE EMISSIONS

The results of the particulate mass emission measurements obtained from the modified Method 5 stack, afterburner, and recuperator tests are given in Table 6.

PCDD and PCDF MEASUREMENTS

The results of the measurements of PCDDs in the stack, afterburner, and recuperator effluent streams are shown in Tables 7 through 10. PCDF results for the same streams are shown in Tables 11 through 14. All samples were analyzed for 2,3,7,8-TCDD and 2,3,7,8-TCDF and for total TCDD and total TCDF. Additionally, a preselected number of samples were analyzed for penta-, hexa-, hepta-, and octa-chlorinated dioxins and furans. The tables also include concentrations, emission rates, and emission factor data (gaseous and particulates) for each test. All the results in the tables have been corrected to 100 percent recovery using the internal standard data.

PCDD and PCDF concentrations found in the reactivated GAC, spent GAC, cyclone catch, and scrubber water samples are shown in Tables 15 and 16, respectively. Total TCDD and TCDF and 2,3,7,8-TCDD and TCDF analysis of each sample type from Test 5 was performed in duplicate. Results of the duplicate analyses are reported and discussed in Section 6 (Quality Assurance Data Summary).

No 2,3,7,8-TCDD was detected in any emission and process streams. TCDD was not detected in any stack filter samples; however, small concentrations were detected in the sorbent (XAD-2) samples of the afterburner and recuperator. The range was 0.006 ng/dscm (0.0004 ppt) to 0.090 ng/dscm (0.006 ppt) in the recuperator and 0.012 ng/dscm (0.008 ppt) in the afterburner. Tetra-CDD was not detected on any stack filter samples; however, small concentrations, 0.012 ng/dscm (0.0008 ppt), were detected in the sorbent (XAD-2) of the afterburner and recuperator, 0.09 ng/dscm (0.006 ppt). Low concentrations, 0.02 ng/g (0.001 ppt), were also found in one cyclone catch sample. Octa-CDD was detected in all emission and process streams, ranging from 3.12 ng/dscm (0.15 ppt) in recuperator particulates to 0.15 ng/g (0.007 ppt) in the spent GAC. Hepta-CDD was detected in all emission streams, ranging from 0.27 ng/dscm (0.014 ppt) in the afterburner particulates to 0.024 ng/dscm (0.001 ppt) in the afterburner XAD-2. Trace amounts of the hepta-CDD were also detected in one spent GAC sample. Penta- and hexa-CDD were detected in the XAD-2 samples of the recuperator, ranging from 0.13 to 0.37 ng/dscm (0.008 to 0.021 ppt), respectively. Trace amounts of hexa-CDD were also detected in the cyclone catch.

TABLE 6. PARTICULATE MASS EMISSION DATA

Parameters	Test No. 1 Recup.	Test No. 2 Recup.	Test No. 3 AB2	Test No. 3 STK	Test No. 4 AB1	Test No. 5 AB2	Test No. 5 STK	Test No. 6 Recup.	Test No. 7 Recup.
Filter Catch Wt., mg	35.3	11,091.3	883.8	31.8	371.6	602.2	32.4	5,796.7	61.4
Rinse Residue Wt., mg	19.7	2,064.9	838.9	3.2	1,047.6	743.4	5.2	4,571.7	51.4
Total Particulate Wt., mg	55.0	13,156.2	1,722.7	35.0	1,419.2	1,345.6	37.6	10,368.4	112.8
Particulate Mass Conc. in Stack Gas, mg/dscm	14.32	2,919.71	550.91	9.72	463.03	427.04	10.56	2,010.55	29.98
Particulate Emission Rate, g/h	20.35	5,219.16	1,277.64	27.31	884.33	825.43	29.25	4,961.53	54.03
Emission Factor,(a) g/kg spent carbon	0.077	19.669	4.815	0.103	3.333	3.111	0.110	18.698	0.204

(a) Assumed spent carbon feed rate of 585 lbs/h (265.35 kg/h).

TABLE 7. PCDD LEVELS IN AFTERBURNER/STACK EMISSIONS - PCDD CONCENTRATION

Test No.	Sample	PCDD Concentration, ng/dscm[a][b]					
		2,3,7,8-	Tetra-	Penta-	Hexa-	Hepta-	Octa-
1-Recup.	Filter	ND	ND (0.050)	ND (0.150)	ND (0.130)	ND (0.270)	3.12
	XAD-2	ND	ND (0.013)	ND (0.007)	ND (0.007)	0.091	0.50
2-Recup.	Filter	ND	ND (0.022)				
	XAD-2	ND	ND (0.010)				
3-AB, 2 sec.	Filter	ND	ND (0.010)				
	XAD-2	ND	ND (0.016)				
3-Stack	Filter	ND	ND (0.008)				
	XAD-2	ND	ND (0.022)				
4-AB, 1 sec.	Filter	ND	ND (0.019)				
	XAD-2	ND	0.012 [3]				
5-AB, 2 sec.	Filter	ND	ND (0.010)	ND (0.004)	ND (0.031)	0.27	0.98
	XAD-2	ND	ND (0.002)	ND (0.007)	ND (0.009)	0.024	0.12
5-Stack	Filter	ND	ND (0.015)	ND (0.007)	ND (0.015)	0.17	1.067
	XAD-2	ND	ND (0.018)	ND (0.023)	ND (0.038)	0.12	0.22
6-Recup.	Filter	NC	ND (0.041)	ND (0.004)	ND (0.031)	0.23	1.38
	XAD-2	ND	0.006 [2]	0.13	0.37	0.23	0.23
7-Recup.	Filter	ND	ND (0.017)				
	XAD-2	ND	0.090 [6]				

(a) ND - not detected. Detection limits in parentheses.
(b) Number of PCDD isomers detected in samples are indicated in brackets.

TABLE 8. PCDD LEVELS IN AFTERBURNER/STACK EMISSIONS - CONCENTRATION IN STACK PARTICULATES

Test No.	Sample	Concentration in Stack Particulates, ng/g[a]					
		2,3,7,8-	Tetra-	Penta-	Hexa-	Hepta-	Octa-
1-Recup.	Filter	ND	ND	ND	ND	ND	220
	XAD-2	ND	ND	ND	ND	6.5	35
2-Recup.	Filter	ND	ND				
	XAD-2	ND	ND				
3-AB, 2 sec.	Filter	ND	ND				
	XAD-2	ND	ND				
3-Stack	Filter	ND	ND				
	XAD-2	ND	ND				
4-AB, 1 sec.	Filter	ND	ND				
	XAD-2	ND	ND				
5-AB, 2 sec.	Filter	ND	ND	ND	ND	0.64	2.3
	XAD-2	ND	ND	ND	ND	0.056	0.28
5-Stack	Filter	ND	ND	ND	ND	16	97
	XAD-2	ND	ND	ND	ND	11	20
6-Recup.	Filter	ND	ND	ND	ND	0.12	0.68
	XAD-2	ND	0.0030	0.064	0.18	0.12	0.12
7-Recup.	Filter	ND	ND				
	XAD-2	ND	3.0				

(a) ND - not detected.

TABLE 9. PCDD LEVELS IN AFTERBURNER/STACK EMISSIONS - EMISSION RATES

Test No.	Sample	Emission Rates, ng/h[a]					
		2,3,7,8-	Tetra-	Penta-	Hexa-	Hepta-	Octa-
1-Recup.	Filter	ND	ND	ND	ND	ND	4,400
	XAD-2	ND	ND	ND	ND	130	700
2-Recup.	Filter	ND	ND				
	XAD-2	ND	ND				
3-AB, 2 sec.	Filter	ND	ND				
	XAD-2	ND	ND				
3-Stack	Filter	ND	ND				
	XAD-2	ND	ND				
4-AB, 1 sec.	Filter	ND	ND				
	XAD-2	ND	23				
5-AB, 2 sec.	Filter	ND	ND	ND	ND	530	1,900
	XAD-2	ND	ND	ND	ND	46	230
5-Stack	Filter	ND	ND	ND	ND	480	3,000
	XAD-2	ND	ND	ND	ND	340	610
6-Recup.	Filter	ND	ND	ND	ND	570	3,400
	XAD-2	ND	15	320	920	570	570
7-Recup.	Filter	ND	ND				
	XAD-2	ND	160				

(a) ND - not detected.

TABLE 10. PCDD LEVELS IN AFTERBURNER/STACK EMISSIONS - EMISSION FACTORS

Test No.	Sample	Emission Factors, ng/kg Spent Carbon(a)					
		2,3,7,8-	Tetra-	Penta-	Hexa-	Hepta-	Octa-
1-Recup.	Filter	ND	ND	ND	ND	ND	17
	XAD-2	ND	ND	ND	ND	0.49	2.7
2-Recup.	Filter	ND	ND				
	XAD-2	ND	ND				
3-AB, 2 sec.	Filter	ND	ND				
	XAD-2	ND	ND				
3-Stack	Filter	ND	ND				
	XAD-2	ND	ND				
4-AB, 1 sec.	Filter	ND	ND				
	XAD-2	ND	0.086				
5-AB, 2 sec.	Filter	ND	ND	ND	ND	2.0	7.2
	XAD-2	ND	ND	ND	ND	0.18	0.88
5-Stack	Filter	ND	ND	ND	ND	1.8	11
	XAD-2	ND	ND	ND	ND	1.3	2.3
6-Recup.	Filter	ND	ND	ND	ND	2.2	13
	XAD-2	ND	0.056	1.2	3.5	2.2	2.2
7-Recup.	Filter	ND	ND				
	XAD-2	ND	0.61				

(a) ND - not detected.

TABLE 11. PCDF LEVELS IN AFTERBURNER/STACK EMISSIONS - PCDF CONCENTRATION

Test No.	Sample	PCDF Concentration, ng/dscm[a][b]					
		2,3,7,8-	Tetra-	Penta-	Hexa-	Hepta-	Octa-
1-Recup.	Filter	ND	0.052 [1]	0.044	ND (0.093)	ND (0.510)	ND (0.950)
	XAD-2	ND	0.094 [2]	ND (0.006)	0.034	0.20	0.17
2-Recup.	Filter	ND	ND (0.039)				
	XAD-2	ND	0.015 [2]				
3-AB, 2 sec.	Filter	ND	ND (0.021)				
	XAD-2	0.015	0.17 [10]				
3-Stack	Filter	ND	ND (0.023)				
	XAD-2	0.018	0.10 [5]				
4-AB, 1 sec.	Filter	ND	ND (0.018)				
	XAD-2	0.049	0.39 [14]				
5-AB, 2 sec.	Filter	ND	ND (0.008)	0.015	0.035	0.073	0.048
	XAD-2	0.021	0.098 [11]	0.044	0.029	0.018	0.012
5-Stack	Filter	ND	ND (0.021)	ND (0.004)	0.0070	0.021	0.018
	XAD-2	ND	ND (0.008)	ND (0.035)	ND (0.050)	ND (0.220)	ND (0.150)
6-Recup.	Filter	ND	ND (0.038)	0.0070	ND (0.012)	ND (0.380)	0.37
	XAD-2	0.016	1.4 [18]	1.1	0.76	0.76	0.35
7-Recup.	Filter	ND	0.010 [1]				
	XAD-2	ND	0.088 [10]				

(a) ND - not detected. Detection limits in parentheses.
(b) Number of PCDF isomers detected in samples are indicated in brackets.

TABLE 12. PCDF LEVELS IN AFTERBURNER/STACK EMISSIONS - CONCENTRATION IN STACK PARTICULATES

Test No.	Sample	Concentration in Stack Particulates, ng/g(a)					
		2,3,7,8-	Tetra-	Penta-	Hexa-	Hepta-	Octa-
1-Recup.	Filter	ND	3.7	3.1	ND	ND	ND
	XAD-2	ND	6.7	ND	2.4	14	12
2-Recup.	Filter	ND	ND				
	XAD-2	ND	0.0050				
3-AB, 2 sec.	Filter	ND	ND				
	XAD-2	0.027	0.30				
3-Stack	Filter	ND	ND				
	XAD-2	1.9	10				
4-AB, 1 sec.	Filter	ND	ND				
	XAD-2	0.11	0.85				
5-AB, 2 sec.	Filter	ND	ND	0.035	0.082	0.17	0.11
	XAD-2	0.049	0.23	0.10	0.068	0.042	0.028
5-Stack	Filter	ND	ND	ND	0.64	1.9	1.6
	XAD-2	ND	ND	ND	ND	ND	ND
6-Recup.	Filter	ND	ND	0.0030	ND	ND	0.18
	XAD-2	0.008	0.68	0.53	0.38	0.38	0.17
7-Recup.	Filter	ND	0.33				
	XAD-2	ND	2.9				

(a) ND - not detected.

TABLE 13. PCDF LEVELS IN AFTERBURNER/STACK EMISSIONS - EMISSION RATES

Test No.	Sample	Emission Rates, ng/h[a]					
		2,3,7,8-	Tetra-	Penta-	Hexa-	Hepta-	Octa-
1-Recup.	Filter	ND	73	63	ND	ND	ND
	XAD-2	ND	130	ND	48	280	250
2-Recup.	Filter	ND	ND				
	XAD-2	ND	27				
3-AB, 2 sec.	Filter	ND	ND				
	XAD-2	35	380				
3-Stack	Filter	ND	ND				
	XAD-2	51	280				
4-AB, 1 sec.	Filter	ND	ND				
	XAD-2	94	750				
5-AB, 2 sec.	Filter	ND	ND	29	68	140	93
	XAD-2	41	190	85	56	35	23
5-Stack	Filter	ND	ND	ND	19	58	50
	XAD-2	ND	ND	ND	ND	ND	ND
6-Recup.	Filter	ND	ND	17	ND	ND	910
	XAD-2	39	3,300	2,600	1,900	1,900	860
7-Recup.	Filter	ND	18				
	XAD-2	ND	160				

(a) ND - not detected.

TABLE 14. PCDF LEVELS IN AFTERBURNER/STACK EMISSIONS - EMISSION FACTORS

Test No.	Sample	Emission Factors, ng/kg Spent Carbon[a]					
		2,3,7,8-	Tetra-	Penta-	Hexa-	Hepta-	Octa-
1-Recup.	Filter	ND	0.28	0.24	ND	ND	ND
	XAD-2	ND	0.50	ND	0.18	1.0	0.93
2-Recup.	Filter	ND	ND				
	XAD-2	ND	0.10				
3-AB, 2 sec.	Filter	ND	ND				
	XAD-2	0.13	1.5				
3-Stack	Filter	ND	ND				
	XAD-2	0.19	1.1				
4-AB, 1 sec.	Filter	ND	ND				
	XAD-2	0.35	2.8				
5-AB, 2 sec.	Filter	ND	ND	0.11	0.26	0.53	0.35
	XAD-2	0.15	0.71	0.32	0.21	0.13	0.087
5-Stack	Filter	ND	ND	ND	0.073	0.22	0.19
	XAD-2	ND	ND	ND	ND	ND	ND
6-Recup.	Filter	ND	ND	0.065	ND	ND	3.4
	XAD-2	0.15	13	9.9	7.0	7.0	3.2
7-Recup.	Filter	ND	0.068				
	XAD-2	ND	0.60				

(a) ND - not detected.

TABLE 15. PCDD LEVELS IN REACTIVATOR PROCESS SAMPLES

Test No.	Sample	PCDD Concentration, ng/g (ng/L)(a)					
		2,3,7,8-	Tetra-	Penta-	Hexa-	Hepta-	Octa-
5	Reactivated GAC	ND	ND	ND	ND	ND	0.28
5	Reactivated GAC	ND	ND				
5	Spent GAC	ND	ND	ND	ND	0.05	0.15
5	Spent GAC	ND	ND				
2	Cyclone Catch	ND	ND				
6	Cyclone Catch	ND	0.02	ND	0.05	ND	0.90
6	Cyclone Catch	ND	ND				
3	Scrubber Water	ND	(ND)				
5	Scrubber Water	ND	(ND)	(ND)	(ND)	(ND)	(0.22)
5	Scrubber Water	ND	(ND)				

(a) ND - not detected.

TABLE 16. PCDF LEVELS IN REACTIVATOR PROCESS SAMPLES

Test No.	Sample	PCDF Concentration, ng/g (ng/L)(a)					
		2,3,7,8-	Tetra-	Penta-	Hexa-	Hepta-	Octa-
5	Reactivated GAC	ND	ND	ND	ND	ND	ND
5	Reactivated GAC	ND	ND				
5	Spent GAC	ND	0.04	ND	ND	0.07	ND
5	Spent GAC	ND	0.10				
2	Cyclone Catch	ND	ND				
6	Cyclone Catch	ND	0.10	ND	0.02	ND	ND
6	Cyclone Catch	0.06	0.77				
3	Scrubber Water	ND	(ND)				
5	Scrubber Water	ND	(ND)	(ND)	(ND)	(ND)	(ND)
5	Scrubber Water	ND	(ND)				

(a) ND - not detected.

No 2,3,7,8-TCDF was detected on the filter samples of any emission stream; however, trace amounts (<0.049 ng/dscm) were found in the XAD-2 samples of all emission streams. Low concentrations (0.06 ng/g) were also observed in one cyclone catch sample. Tetra-CDF was found in recuperator emissions and in the sorbent catch of the afterburner and stack. The range was 0.010 ng/dscm (0.0007 ppt) in recuperator particulate to 1.4 ng/dscm (0.10 ppt) in the vapor phase of the recuperator. Tetra-CDF was also observed in the cyclone catch and in the spent GAC. Penta-CDF was detected in emissions from the afterburner and recuperator. None were detected in stack emissions. The range was 1.1 ng/dscm (0.071 ppt) on the sorbent catch from the recuperator to 0.007 ng/dscm (0.0005 ppt) in particulate samples from the recuperator. No penta-CDF was found in any of the process samples. Hexa-CDF was detected on sorbent samples from the recuperator and afterburner, and on the filters from the afterburner and stack. The range was 0.76 ng/dscm (0.046 ppt) in the recuperator to 0.007 ng/dscm (0.0004 ppt) in the particulate phase of the stack. Trace amounts of the hexa-CDF were also found in the cyclone catch. Hepta-CDF was detected on the sorbent samples from the recuperator and afterburner and on the filters from the afterburner and stack. The range was 0.76 ng/dscm (0.042 ppt) on the sorbent samples from the recuperator to 0.018 ng/dscm (0.001 ppt) on the sorbent samples from the afterburner. Trace amounts (0.07 ng/g (0.004 ppt)) of the hepta-CDF were found in the spent GAC. Octa-CDF was detected on sorbent samples from the recuperator and afterburner and on filter samples from all emission streams. The range was 0.37 ng/dscm (0.018 ppt) on the filter samples from the recuperator to 0.012 ng/dscm (0.0006 ppt) on the sorbent samples from the afterburner. No octa-CDF was detected in any process samples.

Typical HRGC/HRMS selected ion current profiles of the various effluent stream samples are presented in Appendix B. Some samples contained unidentified TCDD isomers present at very low levels relative to the isomers discussed above. When present, 2,3,7,8-TCDF was identified, however other TCDF isomers could not be specifically identified due to lack of suitable reference standards. In general, there does not appear to be any consistent relationship between the isomeric composition of either the TCDDs or the TCDFs in the various samples.

PCBs IN THE FUEL SUPPLY

Sampling for PCBs in the natural gas and fuel oil supply, used to fire the reactivator unit, was performed during modified Method 5 Test No. 1 and No. 7, respectively. The PCB concentration in the extracts of the natural gas (XAD-2) and fuel oil samples was determined by comparison to a standard solution comprised of known quantities of the PCB isomers. The response factors obtained from the standard solution were applied to the sample extract to determine the concentration of all isomers at each level of chlorination. Results of the PCB analyses are shown in Table 17. From these data it can be seen that only one PCB isomer was detected in the natural gas supply and none in the fuel oil. The monochlorobiphenyl (Cl_1) detected in the natural gas was at a very low level (0.71 µg/sample), and just slightly above the detection limit.

TABLE 17. PCB ANALYSIS OF NATURAL GAS AND FUEL OIL[a]

Level of Chlorination	Quantity of PCBs Found, µg				
	Lab. Blank (XAD-2) conc. (D.L.)	Field Blank (XAD-2) conc. (D.L.)	N.G. Sample conc. (D.L.)	F.O. Sample[b] conc. (D.L.)	F.O. Lab. Blank[c] conc. (D.L.)
Cl_1	ND (0.18 µg/sample)	ND (0.18 µg/sample)	0.71 µg/sample	ND (0.17 µg/g)	ND (0.18 µg/g)
Cl_2	ND (0.25 µg/sample)	ND (0.25 µg/sample)	ND (0.25 µg/sample)	ND (0.23 µg/g)	ND (0.25 µg/g)
Cl_3	ND (0.50 µg/sample)	ND (0.50 µg/sample)	ND (0.50 µg/sample)	ND (0.46 µg/g)	ND (0.50 µg/g)
Cl_4	ND (0.71 µg/sample)	ND (0.71 µg/sample)	ND (0.71 µg/sample)	ND (0.66 µg/g)	ND (0.71 µg/g)
Cl_5	ND (0.70 µg/sample)	ND (0.70 µg/sample)	ND (0.70 µg/sample)	ND (0.65 µg/g)	ND (0.70 µg/g)
Cl_6	ND (1.33 µg/sample)	ND (1.33 µg/sample)	ND (1.33 µg/sample)	ND (1.24 µg/g)	ND (1.33 µg/g)
Cl_7	ND (1.36 µg/sample)	ND (1.36 µg/sample)	ND (1.36 µg/sample)	ND (1.26 µg/g)	ND (1.36 µg/g)
Cl_8	ND (2.13 µg/sample)	ND (2.13 µg/sample)	ND (2.13 µg/sample)	ND (1.98 µg/g)	ND (2.13 µg/g)
Cl_{10}	ND (6.80 µg/sample)	ND (6.80 µg/sample)	ND (6.80 µg/sample)	ND (6.30 µg/g)	ND (6.80 µg/g)
Total	ND	ND	0.71	ND	ND

(a) Sample obtained during Test No. 3-Stack.
(b) Based on 1 gram sample.
(c) Sample weight 1.076 grams.

Total sample volume - 1.6 scm (55.061 scf).

PCB concentration in Natural Gas Supply - 0.44 µg/scm (0.013 µg/scf).

TOTAL ORGANIC HALOGENS MEASUREMENTS

Composite reactivated carbon and spent carbon samples obtained during the test program and a sample of virgin carbon of the type being reactivated were analyzed for total organic halogens (TOX) by the Drinking Water Research Division (4), EPA/Cincinnati. Results of the TOX analyses are reported below.

Virgin carbon - <50 mg/Kg
Reactivated carbon - <50 mg/Kg
Spent carbon - 416.3 mg/Kg (average of two samples run in duplicate).

SECTION 5

QUALITY ASSURANCE DATA SUMMARY

During the study, a series of quality assurance (QA) activities were conducted according to a Quality Assurance Project Plan (4). The following sections present a discussion and results of these QA activities.

INSTRUMENT CALIBRATIONS

Modified Method 5 Sampling System

Components of the modified Method 5 train used in the sampling program were calibrated according to the procedures described in the FEDERAL REGISTER (5). The calibration data are presented in Appendix C.

HRGC/HRMS System

The HRGC/HRMS system was calibrated by comparing responses of known quantities of 2,3,7,8-TCDD and 2,3,7,8-TCDF with a known quantity of 2,3,7,8-TCDD-$^{13}C_{12}$ and 2,3,7,8-TCDF-$^{13}C_{12}$, the internal standards. Calibration of the HRGC/HRMS system was checked with a combined 2,3,7,8-TCDD/2,3,7,8-TCDD-$^{13}C_{12}$ standard before and after analysis of each batch of five samples. Typical HRGC/HRMS selected ion current profiles of the 2,3,7,8-TCDD/2,3,7,8-TCDD-$^{13}C_{12}$ standard analyzed with the samples are shown in Appendix B.

All solutions of the 2,3,7,8-TCDD standard were referenced to the same gravimetrically-prepared stock solution. The internal standard material, 2,3,7,8-TCDD$^{13}C_{12}$, however, was obtained in solution. Its concentration has been checked with gravimetrically prepared standards of native (non-isotopically labelled) 2,3,7,8-TCDD from four sources; EPA (R. Harless, HERL/RTP), University of Nebraska, McMaster University, and Monsanto.

The HRMS was tuned to a reference peak from perfluorokerosene (PFK) which was continuously monitored and 2,3,7,8-TCDD peaks were referenced to the PFK peak.

The instrumental response ratio between each TCDD and TCDF isomer observed in the samples and the 2,3,7,8-TCDD-$^{13}C_{12}$ internal standard was measured and the response relative to the internal standard was used to calculate concentration of each TCDD and TCDF isomer.

EXTRACTION EFFICIENCIES

Extraction efficiencies (or recoveries) for the various carbon reactivator samples analyzed during the study are given in Tables 18 and 19. The extraction efficiencies were calculated from the recovery of the 2,3,7,8-TCDD-$^{13}C_{12}$ and 2,3,7,8-TCDF-$^{13}C_{12}$ internal standards, respectively, added to each sample before extraction and HRGC/HRMS analysis.

DUPLICATE ANALYSIS

Results of duplicate analyses of the dryer off-gas cyclone catch, scrubber water, spent carbon feed, and reactivated GAC samples from Tests 5 and 6 are shown in Table 20. Extraction efficiencies are shown in parentheses beside the analytical results. The data are presumed to be more accurate for the determinations in which the higher extraction efficiencies were obtained. Less than (<) values reported in Table 20 are minimum detection limits.

BLANK ANALYSIS

A set of field and laboratory blanks were analyzed along with the carbon reactivator samples. The results of the analysis of the blank samples are presented in Table 21. The Method 5 filter blank was taken from the batch of filters carried to the field site to perform the stack sampling. The methanol/methylene chloride blank combined with the filter blank for analysis was taken from the same batch of reagents and the same Teflon wash bottle used to rinse the components of the Method 5 train. The XAD-2 blank was taken from a set of traps taken to the site to perform the sampling program. The methylene chloride blank combined with the XAD-2 blank for analysis was taken from the same batch of reagent and the same Teflon wash bottle used to rinse the components of the Method 5 vapor phase organic collection module. The benzene and methylene chloride laboratory blanks were taken from the same lot of reagent as that used for extraction of the reactivator samples.

Less than (<) values reported in Table 21 are minimum detection levels for TCDDs and TCDFs in the various reagent and material blanks. Minimum detection levels are higher in the filter and XAD-2 blanks due to interferences present in these materials and are lower in relatively pure solvents, benzene and methylene chloride, which were used for the sample extractions.

ACCURACY AND PRECISION

Estimates of accuracy and precision of the TCDD measurements were made by spiking XAD-2 traps from Test 5 with known quantities of 1,2,3,4-TCDD$^{13}C_{12}$. The 1,2,3,4-TCDD$^{13}C_{12}$ isomer was selected for spiking because this labelled isomer is not present in combustion sources. The XAD-2 traps were spiked prior to conducting the sampling and thus would reflect errors in the TCDD measurements due to volatilization or decomposition losses during sampling as

TABLE 18. EXTRACTION EFFICIENCIES FOR CARBON REACTIVATOR SAMPLES BASED ON RECOVERY OF 2,3,7,8-TCDD$^{13}C_{12}$

Test No.	MM5 Recuperator		MM5 Stack		MM5 Afterburner(a)		Cyclone Catch	Scrubber Water	Spent GAC	Reactivated GAC
	Front	Back	Front	Back	Front	Back				
1	104	125	-(b)	-	-	-	-	-	-	-
2	63	123	-	-	-	-	50	-	-	-
3	-	-	108	102	139	123	-	57	-	-
4	-	-	-	-	23	79	-	-	-	-
5	-	-	52	70	53	89	-	74	67	151
6	54	83	-	-	-	-	46	-	-	-
7	59	98	-	-	-	-	-	-	-	-

(a) Afterburner was sampled at Sec. 1 for Test 4, and at Sec. 2 for Tests 3 and 5.
(b) Dash indicates that location was not sampled.

TABLE 19. EXTRACTION EFFICIENCIES FOR CARBON REACTIVATOR SAMPLES BASED ON RECOVERY OF 2,3,7,8-TCDF $^{13}C_{12}$

Test No.	MM5 Recuperator		MM5 Stack		MM5 Afterburner(a)		Cyclone Catch	Scrubber Water	Spent GAC	Reactivated GAC
	Front	Back	Front	Back	Front	Back				
1	91	86	-(b)	-	-	-	-	-	-	-
2	64	103	-	-	-	-	51	-	-	-
3	-	-	90	79	117	109	-	50	-	-
4	-	-	-	-	27	80	-	-	-	-
5	-	-	45	75	77	85	-	73	69	75
6	55	81	-	-	-	-	48	-	-	-
7	51	96	-	-	-	-	-	-	-	-

(a) Afterburner was sampled at Sec. 1 for Test 4, and at Sec. 2 for Tests 3 and 5.
(b) Dash indicates that location was not sampled.

TABLE 20. DUPLICATE ANALYSIS RESULTS

Analyte	Concentration (Extraction Efficiency)[a] of Analyte in Sample, pg/g (%)			
	Cyclone Catch (Test No. 6)	Scrubber Water (Test No. 5)	Spent GAC (Test No. 5)	Reactivated GAC (Test No. 5)
2,3,7,8-TCDD	<7.9 (46) <9.4 (55)	<0.002 (74) <0.011 (53)	<6.4 (67) <4.8 (77)	<25 (151) <29 (118)
Total TCDD	21 (46) <9.4 (55)	<0.002 (74) <0.011 (53)	<6.4 (67) <4.8 (77)	<25 (151) <29 (118)
2,3,7,8-TCDF	<18 (48) 61 (57)	<0.003 (73) <0.026 (50)	<8.4 (69) <4.9 (73)	<38 (75) <34 (84)
Total TCDF	98 (48) 770 (57)	<0.003 (73) <0.026 (50)	39 (69) 100 (73)	<38 (75) <34 (84)

(a) Extraction efficiencies based on recovery of 2,3,7,8-TCDD-$^{13}C_{12}$ for 2,3,7,8-TCDD and total TCDD results, and on recovery of 2,3,7,8-TCDF-$^{13}C_{12}$ for 2,3,7,8-TCDF and total TCDF results.

TABLE 21. BLANK ANALYSIS RESULTS

Blank	TCDD Concentration, pg/sample		TCDF Concentration, pg/sample	
	2,3,7,8	Total	2,3,7,8	Total
Lab Blank (GAC)	<17[a]	<17[a]	<48[a]	<48[a]
MM5 Front Blank	<4.5	<4.5	<5.3	<5.3
Lab Blank (MM5)	<40	<40	<150	<150
MM5 Back Blank	<20	<20	<23	<23
Lab Blank	<5.6	<5.6	<4.4	19
Lab Blank (SW)	<0.007[a]	<0.007[a]	<0.009[a]	<0.009[a]
Lab Blank (GAC Fines)	<9.8[a]	25[a]	<18[a]	<18[a]

(a) Concentration in units of pg/g, rather than pg/sample.

well as errors occuring during the analysis steps. In addition to the XAD-2 field spikes, scrubber water and GAC fines were spiked in the laboratory with 2,3,7,8-TCDD and 2,3,7,8-TCDF.

Duplicate analyses of the cyclone catch, scrubber water, spent carbon feed, and reactivated GAC samples from Tests 5 and 6 were also performed to provide an estimate of the precision of the TCDD and TCDF measurements. Duplicate analyses of the Method 5 samples were not performed because the samples (filter and XAD-2) could not be accurately subdivided.

The accuracy (i.e., percent recovery) and precision estimates obtained from the spike and duplicate analyses are shown in Tables 22, 23, and 24. The accuracy values reported in Tables 22 and 23 were calculated by the equation:

$$\text{Accuracy, \%} = \frac{(X)\ 100}{T}$$

where X is the concentration found by analysis and T is the quantity of spike added.

The precision estimates were obtained by calculating the absolute standard deviation (n-1) for the two determinations for each sample type. The precision estimates are also given as the relative standard deviation (RSD) which was calculated by the equation

$$\text{RSD, \%} = \frac{\sigma 100}{\bar{x}}$$

where σ is the absolute standard deviation and $\bar{x}$ is the average quantity of 1,2,3,4-TCDD found for the two determinations.

The precision estimates obtained from the duplicate TCDD and TCDF analyses are shown in Table 24. Only two samples had detectable levels of analyte in both duplicates. The absolute standard deviations (n-1) were calculated from the two determinations of the 2,3,7,8- and total TCDD and the 2,3,7,8- and total TCDF for each sample type. The RSD was calculated as described previously.

In general, the accuracy and precision estimates obtained from the 1,2,3,4-TCDD-$^{13}C_{12}$ spike data are well within the quality assurance objectives for the program. The poorer precision estimated from some of the duplicate TCDD and TCDF analyses is due to the increased variability associated with measurement of concentrations approaching the detection limit.

COMPLETENESS

Valid results are reported for all samples for which collection and analyses were planned during this study. Therefore, the completeness objective of 100 percent was achieved.

TABLE 22. LABORATORY BLANK SPIKE DATA

Sample Type	2,3,7,8-TCDD Spike			2,3,7,8-TCDF Spike		
	Amount Added, pg/g	Amount Found, pg/g	Accuracy, %	Amount Added, pg/g	Amount Found, pg/g	Accuracy, %
Scrubber Water	5	5.1	102	5	5.7	114
GAC Fines	25,000	25,000	100	25,000	26,000	104

TABLE 23. FIELD SPIKE DATA

MM5 Sampling Location	1,2,3,4-TCDD$^{13}C_{12}$ Added, ng	1,2,3,4-TCDD$^{13}C_{12}$ Found, ng	Accuracy, %
Stack	2.5	2.5	100
Afterburner	2.5	2.1	84

Average Spike Found, ng - 2.29
Average Accuracy, % - 92
Standard Deviation (σ) - 0.30
RSD, % - 13.1

TABLE 24. DUPLICATE ANALYSIS DATA

Sample	Analyte	Amount Found, pg/g	Average, pg/g	Std. Dev. (σ)	RSD, %
Cyclone Catch (Test No. 6)	Total TCDF	98 770	434	475	109
Spent GAC (Test No. 5)	Total TCDF	39 100	69.5	43	62

DATA RECORDS

Data accumulated during this study are recorded in Battelle-Columbus Laboratories (BCL) Laboratory Record Books Nos. 39874 and 37900. The mass spectral data are recorded on magnetic tapes and stored in the BCL Mass Spectrometry Laboratory tape library.

The magnetic tape records will be retained for three years following completion of the study. The Laboratory Records Books will be retained in a permanent file maintained by Battelle-Columbus Laboratories.

REFERENCES

1. Howes, J. E., Jr., DeRoos, F. L., Aichele, D., Kohler, D. F., and Larson, M. E. "Determination of Dioxin Levels in Carbon Reactivation Process Effluent Streams". Final Report on EPA Contract No. 68-02-3487, Work Assignment No. 16, May 1983.

2. Stanley, J. S., et al., "Sampling and Analysis Protocol for Assessing Organic Emissions from Stationary Combustion Sources in Exposure Evaluation Division Studies", EPA-560/5-82-014 (January 1982).

3. Kooke, R.M.M., Lustenhouwer, J.W.A., Olie, K., and Hutzinger, O., "Extraction Efficiencies of Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans from Fly Ash", Analytical Chemistry, 53, No. 3, pp. 461-463 (March 1981).

4. Method 450.1 - Determination of Total Organic Halogens, USEPA, Environmental and Support Laboratory.

5. Quality Assurance Project Plan for Determination of Dioxin and Furan Levels in Effluent Streams of the Cincinnati Waterworks Carbon Reactivation System, Battelle-Columbus Laboratories (December 28, 1984).

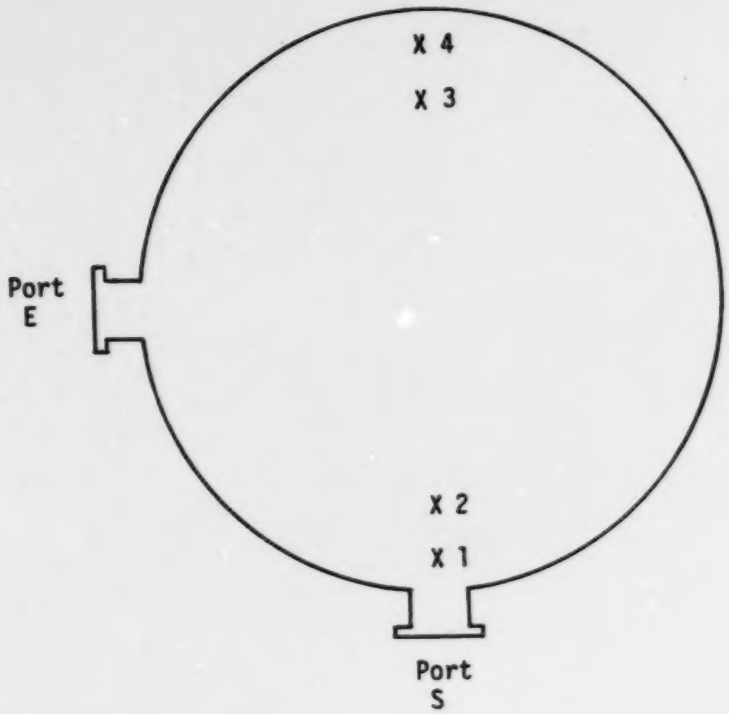
6. Method 5 - Determination of Particulate Emissions From Stationary Sources, Federal Register, Vol. 42, No. 160--Thursday, August 18, 1977.

APPENDIX A

FIELD SAMPLING AND SAMPLE RECOVERY DATA

The field data recorded in the collection of samples during Test No. 3 from the Cincinnati Waterworks reactivator system are presented in Figures A-1 through A-9. The order used to present the figures are:

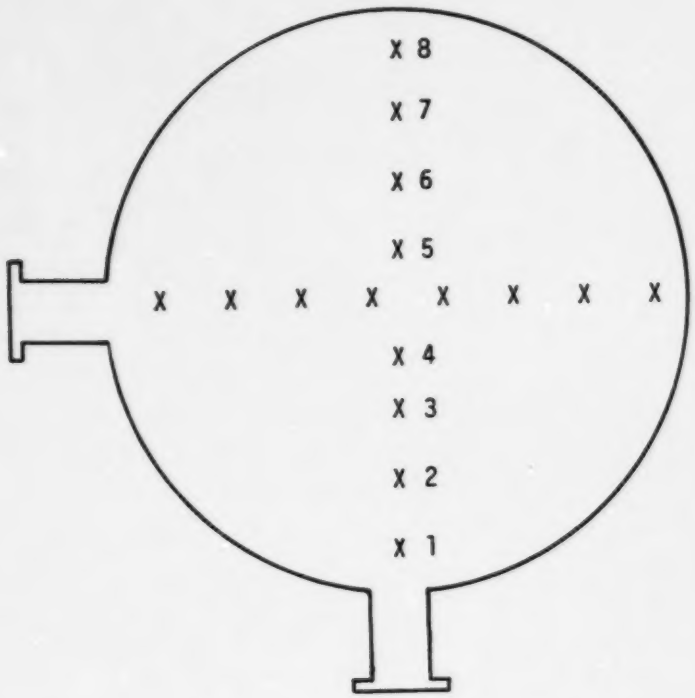
- Velocity and temperature traverse data - stack and recuperator
- Modified Method 5 system operating data
- Fyrite gas measurements for CO_2 and O_2
- Modified Method 5 sample recovery data
- Reactivated GAC, spent GAC, cyclone catch, and scrubber water sampling data



STACK ID = 8.5 in. (21.6 cm)

Traverse Point	Distance from Port on ID, in (cm)	Velocity Head (ΔP) in H_2O (cm H_2O)	Stack Temperature (T_s) °F	(°C)
S-1	0.57 (1.45)	1.23 (3.12)	116	(46.7)
S-2	2.13 (5.41)	1.35 (3.49)	116	(46.7)
S-3	6.38 (16.21)	1.60 (4.06)	116	(46.7)
S-4	7.93 (20.14)	1.55 (3.94)	116	(46.7)
Average		1.43 (3.65)	116	(46.7)

Figure A-1. Stack velocity and temperature traverse data.



RECUPERATOR ID = 20 in. (51 cm)

Traverse Point	Distance from Port on ID, in (cm)	Velocity Head (ΔP) in H_2O (cm H_2O)	Stack Temperature (T_s) °F	(°C)
1	0.7 (1.78)	0.14 (0.36)	1131	(610)
2	2.1 (5.33)	0.12 (0.30)	1135	(613)
3	3.9 (9.91)	0.11 (0.28)	1139	(615)
4	6.5 (16.51)	0.09 (0.23)	1141	(616)
5	13.5 (34.29)	0.07 (0.18)	1144	(618)
6	16.1 (40.89)	0.08 (0.20)	1147	(619)
7	17.9 (45.47)	0.09 (0.23)	1147	(619)
8	19.3 (49.02)	0.09 (0.23)	1127	(608)
Average		0.099 (0.25)	1138	(615)

Figure A-2. Recuperator velocity and temperature traverse data.

Plant/Address: Cincinnati Waterworks
Date: 12/8/84
Sampling Location: Regenerator Stack
Source Code: CW-2
Run Number: BCL-CW-120884-MM5-3
Operator: D. Kohler
Ambient Temperature, °F: 80
Barometric Pressure, in. Hg: 29.50
Static Pressure, in. Hg: +0.32

Pre-Test Leak Rate
0.003 cfm @ 15 in. Hg
Post-Test Leak Rate
0.002 cfm @ 8 in. Hg

Pitot tubes checked at >2" H_2O before test

Probe Length and Type: 4' Pyrex S/N:
Nozzle S/N: Nozzle ID: 0.155
Assumed Moisture, %: 9
XAD-2 Module S/N: 600
Meter Box S/N: 1970 Oven S/N:
Meter $\Delta H_@$: 1.9
C Factor: 1.0
Probe Heater Setting: 250
Oven Setting: 250
Reference ΔP:
Calculations Checked By: J. Howes Date:

Traverse Point Number	Clock Time (24-hr Clock) / Sampling Time, min		Gas Meter Reading, ft^3	Velocity Head, in. H_2O	Orifice Pressure Differential, in. H_2O		Stack Temperature, °F	Dry Gas Meter Temperature		Pump Vacuum, in. Hg	Oven Temperature, °F	Impinger Temperature, °F	Sorbent Trap Temperature, °F	Probe Temperature, °F
					Desired	Actual		Inlet, °F	Outlet, °F					
E-1	0	1417	911.887	2.00	1.18	1.80	98	90	90	6	247	42	54	269
	15	1432	922.225	2.00	1.18	1.18	99	112	93	6	241	45	48	252
	30	1447	930.363	"	"	"	101	118	99	5.5	241	49	50	261
	45	1502	938.928	"	"	"	103	124	104	5.5	241	49	49	260
	60	1517	947.590	"	"	"	101	128	108	5.5	241	54	49	256
	75	1532	956.257	"	"	"	101	129	111	5.5	240	52	48	255
	90	1547	964.900	"	"	"	102	130	113	5.5	240	53	49	254
	105	1602	973.625	"	"	"	101	131	115	6.0	240	54	51	258
	120	1617	982.355	"	"	"								
	(Sampling stopped for 73 minutes - changing ports in afterburner test)													
	120	1730	982.355	2.00	1.18	1.18	104	100	101	6.5	236	65	63	295
	135	1745	991.037	"	"	"	104	116	102	6.5	238	50	49	265
	150	1800	999.625	"	"	"	105	124	108	6.5	233	49	49	257
	165	1815	1008.230	"	"	"	105	128	109	7.0	227	50	49	253
	180	1830	1016.910	"	"	"	104	129	112	7.0	237	53	50	253
	195	1845	1025.615	"	"	"	104	130	113	7.0	233	56	52	253
	210	1900	1034.280	"	"	"	104	131	114	7.3	237	57	53	253
	225	1915	1043.100	"	"	"	104	131	114	7.3	201	55	50	253
	240	1930	1051.780											
Total	240		139.893	$(\sqrt{\Delta P})$Avg										
Average				1.414	1.18		102.5	114.3			236		51	259

Figure A-3. Modified Method 5 stack sampling data - Test No. 3 STK.

Date 12/8/84 Plant Cincinnati Waterworks

Run No. BCL-CW-120884-MM5-3 Sampling Location Carbon Regenerator Stack

Performed by D. Kohler Sample Type Fyrite Grab

Time	CO_2 Concentration, % by Volume, dry	O_2 Concentration, % by Volume, dry	N_2 Concentration,* % by Volume, dry
1453	13	5	
1555	12	6	
1807	14	4	
1853	12	5	
Average	12.75	5	82.25

Figure A-4. Fyrite analysis of stack gas - Test No. 3 STK.

Plant/Location Cincinnati Waterworks

Sampling Location Carbon Regenerator Stack

Train Type Mod. Method 5 Run No. BCL-CW-120884-MM5-3 Run Date 12/8/84

Recovery Date 12/8/84 Recovered by Howes/Kohler Checked by J. Howes

1.0 CYCLONE CATCH DATA

Cyclone	Sample No.	Tare, g	Gross, g	Net, g
10 µm		N/A		
3 µm				
1 µm				

2.0 FILTER CATCH DATA

Filter Size/Material 3" diameter Pallflex 2500 quartz filter

Filter No.	Sample No.	Tare, g	Gross, g	Net, g
PD-3	BCL-CW-120884-MM5-3 STK QPF	0.3408	0.3726	0.0318

Filter Total 0.0318 g

Figure A-5. Modified Method 5 sample recovery data - Test No. 3 STK.

3.0 CONDENSATE COLLECTION

Total Condensate Collected _____ g

185 ml

4.0 IMPINGER DATA

Impinger No.	Sample No.	Loading	Initial*	Final*	Gain*
1	discarded	100 ml DI water	562.5	570.0	7.5
2	"	"	577.9	580.7	2.3
3	"	empty	533.2	535.4	2.2
4					
5					

Total Gain* 12 grams

* Indicate whether ml or g

5.0 SILICA GEL WEIGHTS

Initial Weight, g	Final Weight, g	Weight Gain, g
842.7	872.3	29.6

Total Weight Gain 29.6 g

6.0 TOTAL MOISTURE COLLECTED

Total Collected = Condensate, g + Total Imp. Gain, g + Total Silica Gel Gain, g

= 226.6 g

7.0 SOLID SORBENT TRAP

Solid Sorbent Material XAD-2

Weight Used 22 g g

Trap No. 600

Sample No. BCL-CW-120884-MM5-3 STK-XAD

Figure A-5. (Continued).

8.0 RINSE SOLUTION DATA

Component	Sample No.	Rinse Composition	Rinse Volume, ml
Front Half	BCL-CW-120884-MM5-3-FHR	MeOH/MeCl 50:50	170
Condenser Mod.	BCL-CW-120884-MM5-3-CMR	MeCl	not measured
Impinger #1			
Impinger #2			
Impinger #3			
Impinger #4			

Comments: (Indicate condition of filters, solution colors, presence of precipitates, odor, etc.)

Filter in good condition; no evidence of leakage.
Light gray deposit on filter.
Condensate clear
Impinger solutions clear

Front half rinse residue (rinse volume = 170 ml)

G 111.6309 g
T 111.6277 g
N 0.0032 g

Blank correction negligible

Net residue wt = 0.0032 g
Net filter wt = 0.0318
Total net particulate wt = 0.0350 g

Figure A-5. (Continued).

Plant Cincinnati Waterworks — Sampling Location Drop Chute to Quench Tank

Date 12/8/84 — Sampling Method Grab (200 ml beaker)

Sampled By D. Hartman — Test No. BCL-CW-120884-MM5-3

Process Steam ID Reactivated GAC (RC) — Composite or Single Sample Composite

Sample Description* Ahead of Quench Tank
Test started at 1417 hours

Time Sample Taken	Sample Number	Sample Weight/Volume	Observations
1430	BCL-CW-120884-RC-3-1	88.7 g 200 ml	Composited
1500	" 2	89.7 g "	"
1530	" 3	87.8 g "	"
1600	" 4	79.3 g "	"
1800	" 5	92.8 g "	"
1830	" 6	94.3 g "	"
1900	" 7	94.9 g "	"
1930	" 8	95.1 g "	"
	BCL-CW-120884-RC-3-C	284.3 g 1600 ml	Composite of above samples

Figure A-6. Reactivated GAC sampling data - Test No. 3 STK.

Plant Cincinnati Waterworks — Sampling Location Drop Chute from Screw Feeder

Date 12/8/84 — Sampling Method Grab and Composite

Sampled By D. Hartman — Test No. BCL-CW-120884-MM5-3

Process Steam ID SC (Spent GAC) — Composite or Single Sample Composite

Sample Description* Spent Carbon Feed, Wet

Test started at 1417 hours

Time Sample Taken	Sample Number	Sample Weight/Volume	Observations
1430	BCL-CW-120884-SC-3-1	233.2 g 200 ml	Composited
1500	" 2	237.3 g "	"
1530	" 3	209.6 g "	"
1600	" 4	238.3 g "	"
1800	" 5	252.8 g "	"
1830	" 6	242.5 g "	"
1900	" 7	236.0 g "	"
1930	" 8	247.9 g "	"
	BCL-CW-120884-SC-3-C	1897.6 g 1600 ml	Composited from above samples

Figure A-7. Spent GAC sampling data - Test No. 3 STK.

Plant Cincinnati Waterworks | Sampling Location Drop-Out Drum

Date 12/8/84 | Sampling Method Grab

Sampled By D. Hartman | Test No. BCL-CW-120884-MM5-3

Process Steam ID CC (Cyclone Catch) | Composite or Single Sample Composite

Sample Description* Fine carbon separated by cyclone from dryer off-gas stream

Test started at 1417 hours

Time Sample Taken	Sample Number	Sample Weight/Volume	Observations
1430	BCL-CW-120884-CC-3-1	~100 g 200 ml	Composite
(Only one grab sample of cyclone catch was obtained due to problems at the sampling area.)			

Figure A-8. Cyclone catch sampling data - Test No. 3 STK.

Plant Cincinnati Waterworks
Date 12/8/84
Sampled By D. Hartman
Process Steam ID SL (Scrubber Liquid)
Sample Description* Combined effluent from Venturi and tray scrubber
Test started at 1417 hours

Sampling Location Tap in Discharge Line
Sampling Method Grab and Composite
Test No. BCL-CW-120884-MM5-3
Composite or Single Sample Composite

Time Sample Taken	Sample Number	Sample Weight/Volume	Observations
1430	BCL-CW-120884-SL-3-1	16 oz (~480 ml)	Composited
1500	" 2	" "	"
1530	" 3	" "	"
1600	" 4	" "	"
1800	" 5	" "	"
1830	" 6	" "	"
1900	" 7	" "	"
1930	" 8	" "	"
	BCL-CW-120884-SL-3-C	1 gallon	Composite of above samples

Figure A-9. Scrubber water sampling data - Test No. 3 STK.

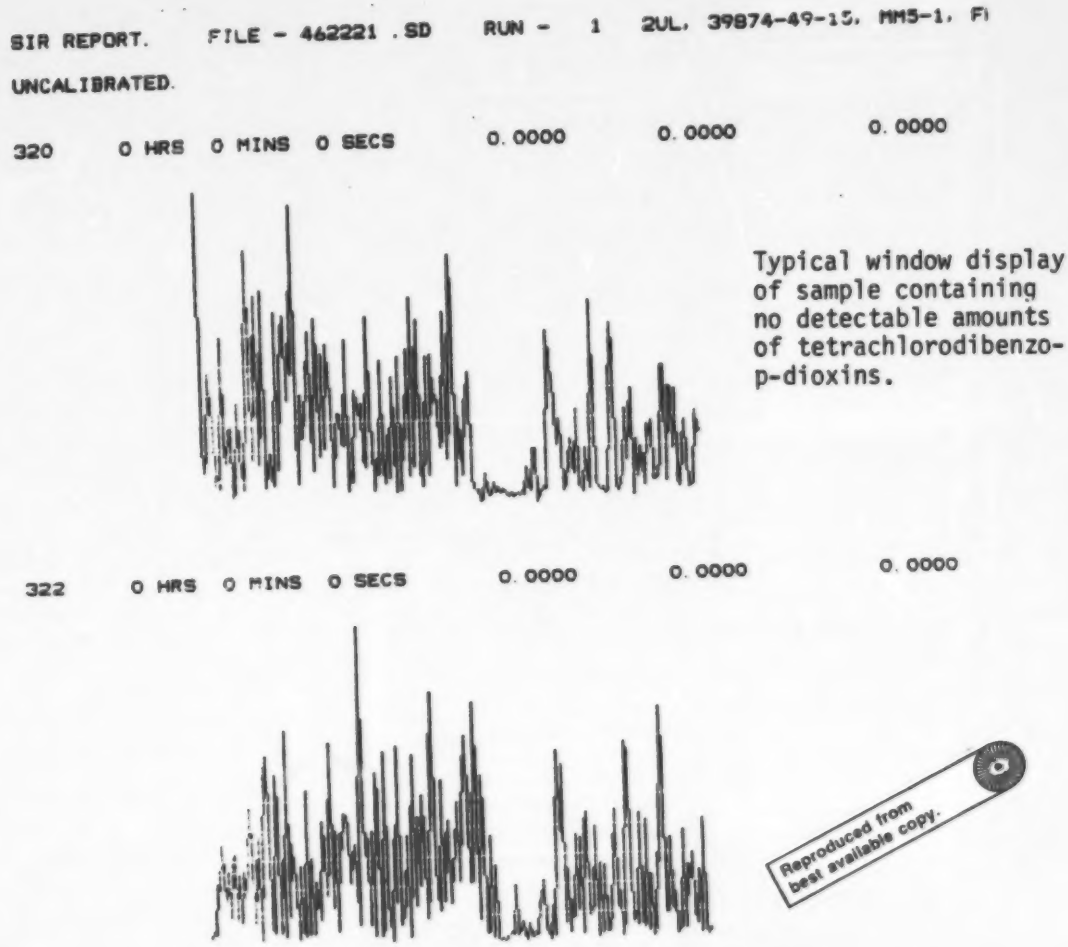
APPENDIX B

MASS SPECTRA DATA

Typical HRGC/HRMS selected ion current profiles for PCDDs, PCDFs, and the internal standards are shown in Figures B-1 through B-32. These data were obtained from the MM5 front and back samples collected in Test 1. In all PCDD and PCDF selected ion current profiles, the most intense peak has been normalized to full scale. The exact masses monitored for acquisition of these profiles are shown in Table B-1.

TABLE B-1. EXACT MASSES USED FOR THE DETERMINATION OF PCDDs AND PCDFs

Compound	Accurate Mass		Theoretical Isotope Ratio
	Mass 1	Mass 2	Mass 1/Mass 2
Tetrachlorodibenzo-p-dioxins	319.8965	321.8936	0.77
Tetrachlorodibenzofurans	303.9016	305.8987	0.77
Pentachlorodibenzo-p-dioxins	355.8546	357.8517	1.54
Pentachlorodibenzofurans	339.8597	341.8567	1.54
Hexachlorodibenzo-p-dioxins	389.8156	391.8127	1.23
Hexachlorodibenzofurans	373.8207	375.8178	1.23
Heptachlorodibenzo-p-dioxins	423.7766	425.7737	1.03
Heptachlorodibenzofurans	407.7817	409.7788	1.03
Octachlorodibenzo-p-dioxin	457.7377	459.7347	0.88
Octachlorodibenzofuran	441.7428	443.7398	0.88


SIR REPORT. FILE - 462221 .SD RUN - 1 2UL, 39874-49-13, MM5-1, Fl
UNCALIBRATED.
320 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000
322 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000
Test No. 1 - MM5 Front Sample
Column - 30m DB-5 Fused Silica

Typical window display of sample containing no detectable amounts of tetrachlorodibenzo-p-dioxins.

Reproduced from best available copy.

Figure B-1. HRGC/HRMS selected ion current profile for tetrachlorodibenzo-p-dioxins.

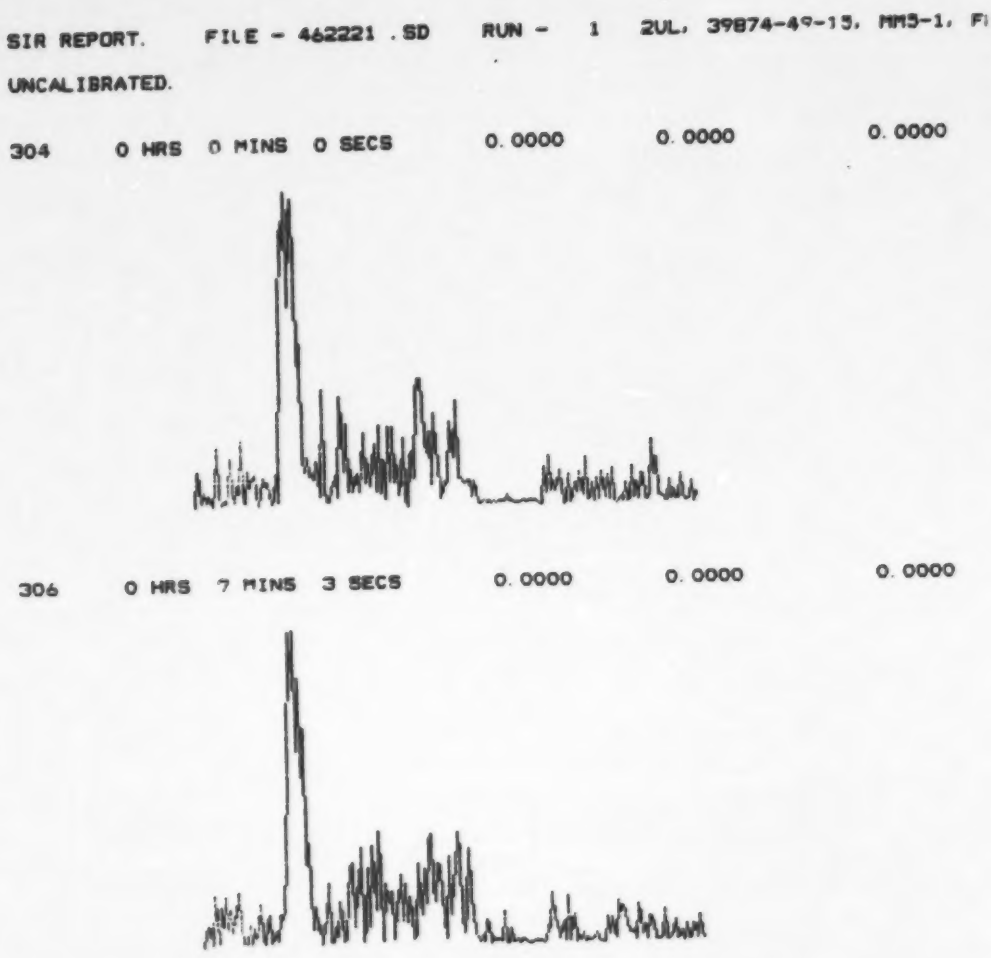

SIR REPORT. FILE - 462221 .SD RUN - 1 2UL, 39874-49-15, MM5-1, FI
UNCALIBRATED.
304 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000
306 0 HRS 7 MINS 3 SECS 0.0000 0.0000 0.0000
Test No. 1 - MM5 Front Sample
Column - 30m DB-5 Fused Silica

Figure B-2. HRGC/HRMS selected ion current profile for tetrachlorodibenzoforans.

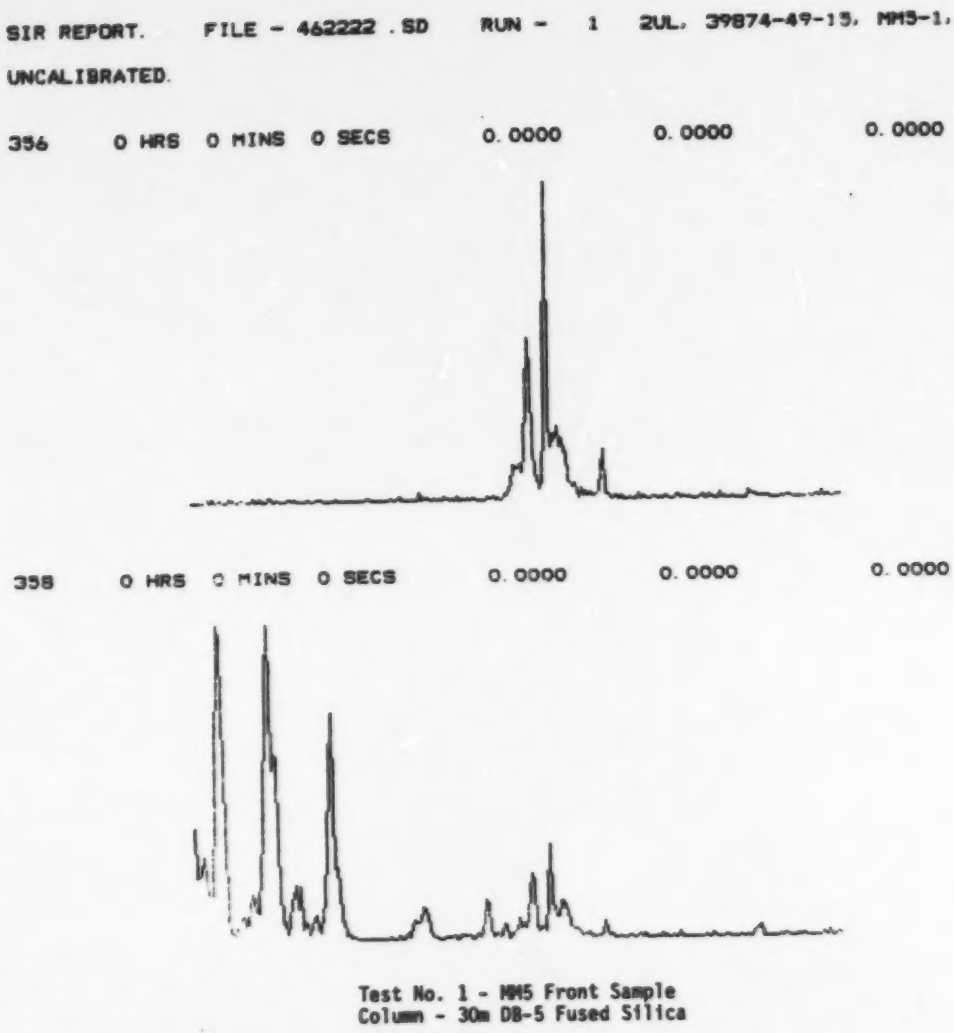

SIR REPORT. FILE - 462222 .SD RUN - 1 2UL. 39874-49-15, MM5-1, FI
UNCALIBRATED.
356 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000
358 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000
Test No. 1 - MM5 Front Sample
Column - 30m DB-5 Fused Silica

Figure B-3. HRGC/HRMS selected ion current profile for pentachlorodibenzo-p-dioxins.

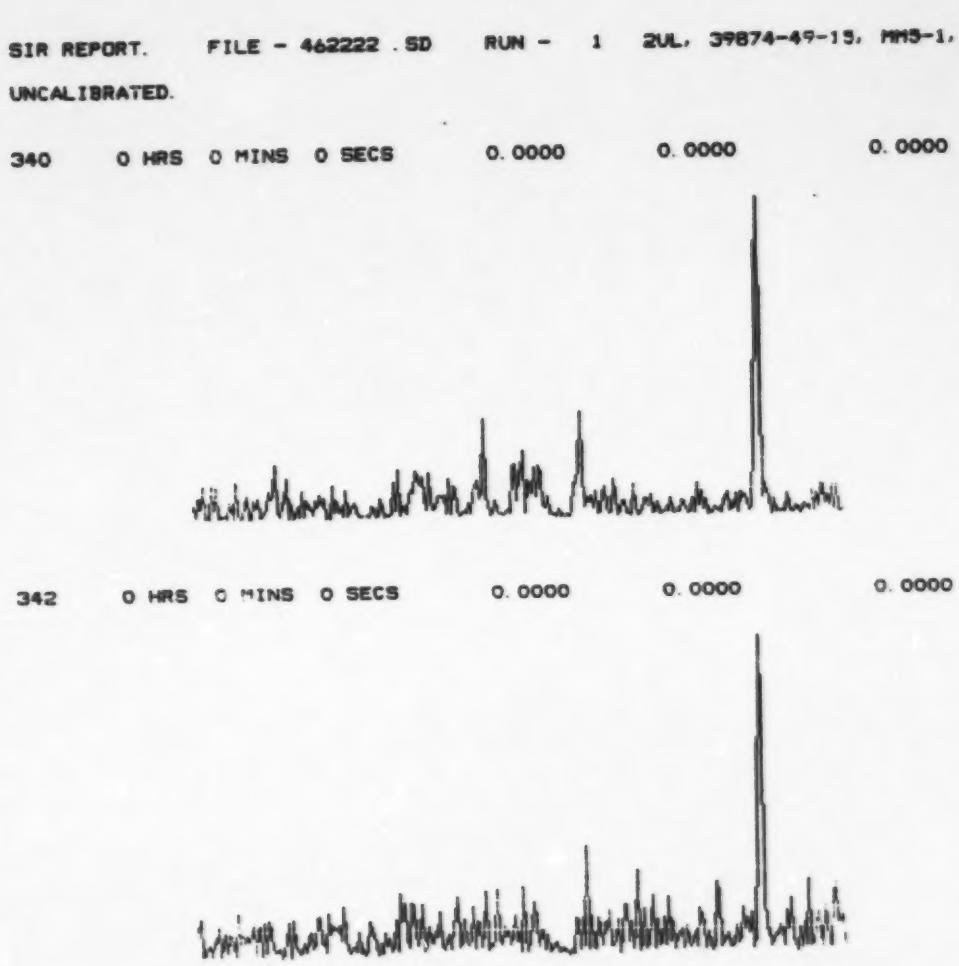

SIR REPORT. FILE - 462222 .SD RUN - 1 2UL, 39874-49-15, MM5-1, Fl
UNCALIBRATED.
340 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000
342 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000
Test No. 1 - MM5 Front Sample
Column - 30m DB-5 Fused Silica

Figure B-4. HRGC/HRMS selected ion current profile for pentachlorodibenzofurans.

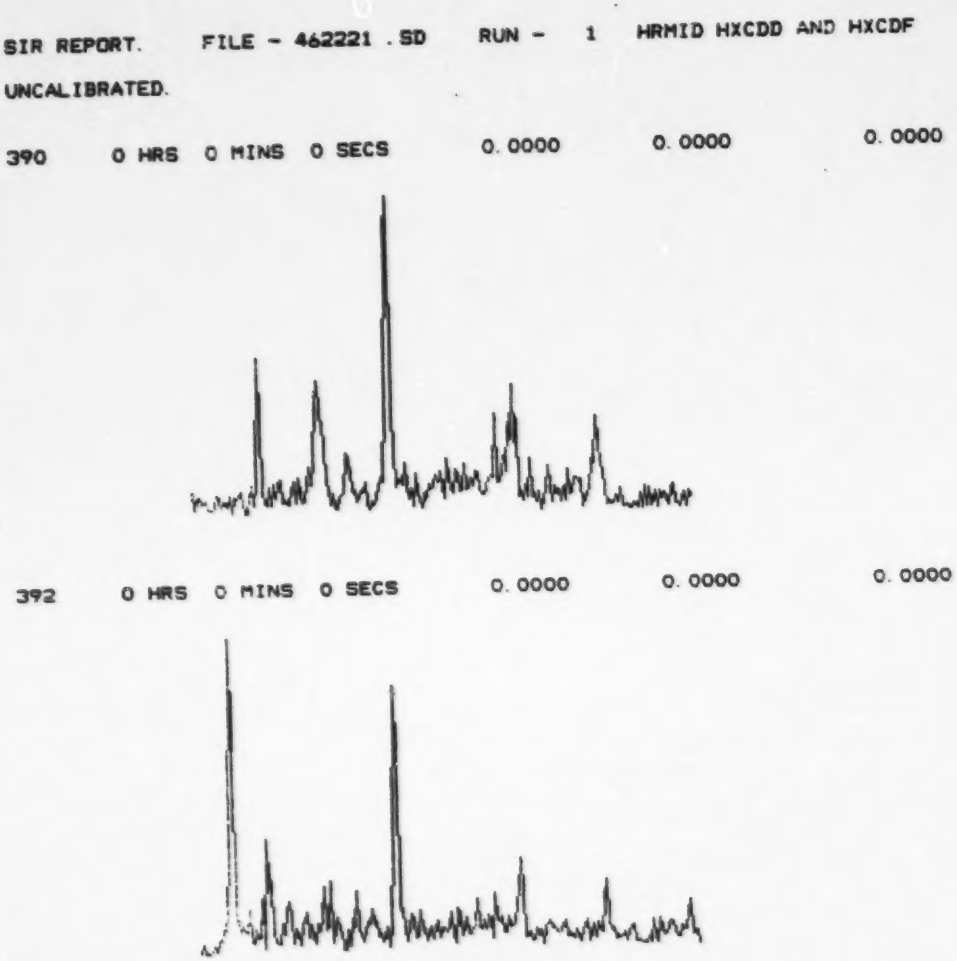

SIR REPORT. FILE - 462221 .SD RUN - 1 HRMID HXCDD AND HXCDF
UNCALIBRATED.
390 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000
392 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000
Test No. 1 - MM5 Front Sample
Column - 30m DB-5 Fused Silica

Figure B-5. HRGC/HRMS selected ion current profile for hexachlorodibenzo-p-dioxins.

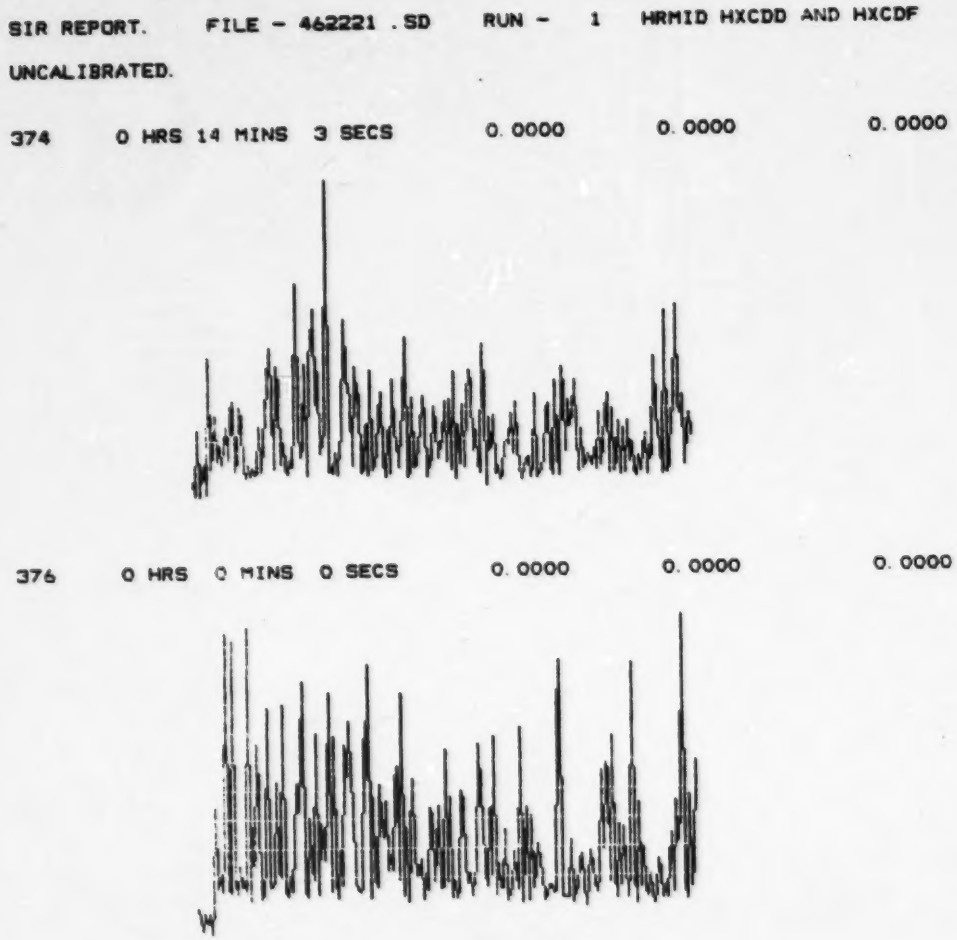
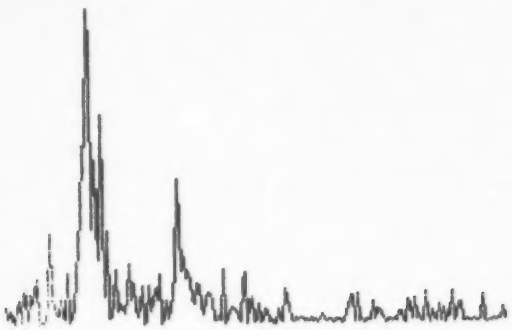

SIR REPORT. FILE - 462221 .SD RUN - 1 HRMID HXCDD AND HXCDF
UNCALIBRATED.
374 0 HRS 14 MINS 3 SECS 0.0000 0.0000 0.0000
376 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000
Test No. 1 - MM5 Front Sample
Column - 30 m DB-5 Fused Silica

Figure B-6. HRGC/HRMS selected ion current profile for hexachlorodibenzofurans.

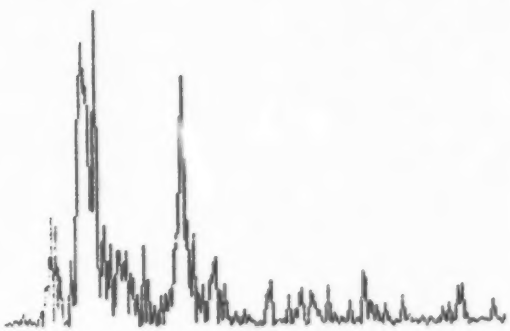
SIR REPORT. FILE - 462222 .SD RUN - 1 HRMID HEPTA-CDF + HEPTA-CD.

UNCALIBRATED.

424 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000



426 0 HRS 10 MINS 2 SECS 0.0014 0.0014 0.0000



Test No. 1 - MM5 Front Sample
Column - 30m DB-5 Fused Silica

Figure B-7. HRGC/HRMS selected ion current profile for heptachlorodibenzo-p-dioxins.

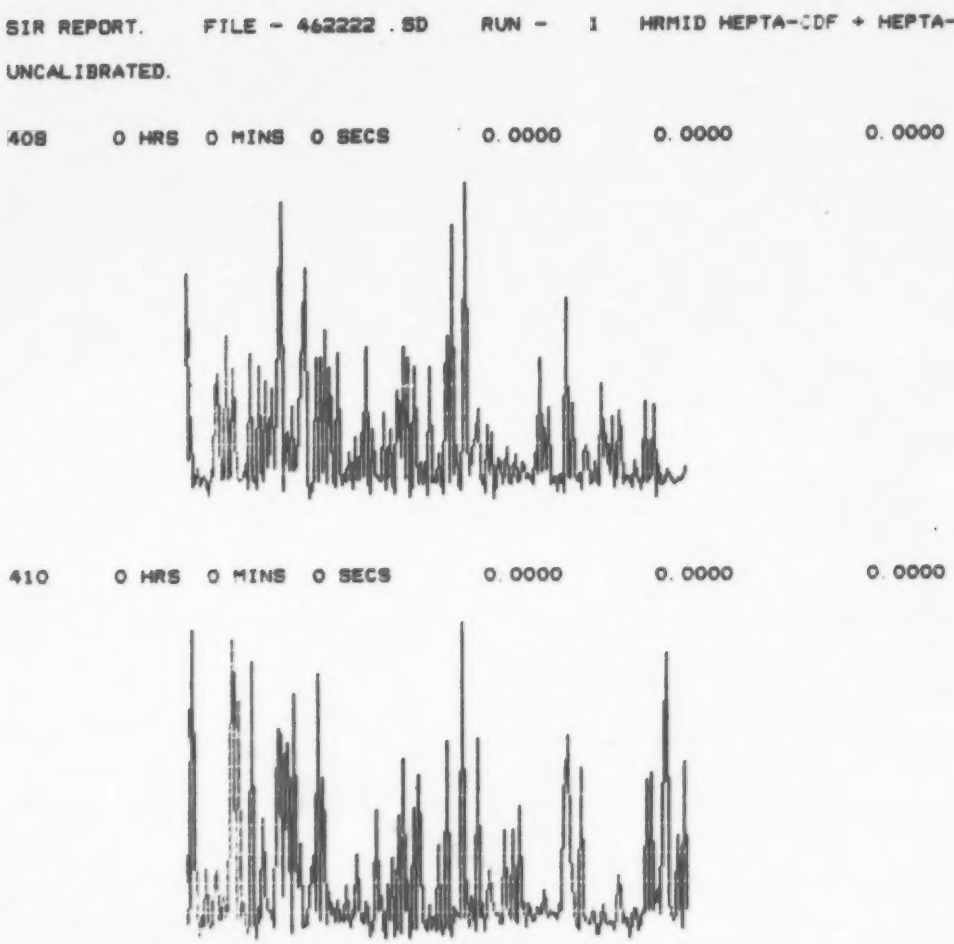

SIR REPORT. FILE - 462222 .SD RUN - 1 HRMID HEPTA-CDF + HEPTA-CD
UNCALIBRATED.
408 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000
410 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000
Test No. 1 - MM5 Front Sample
Column - 30m DB-5 Fused Silica

Figure B-8. HRGC/HRMS selected ion current profile for heptachlorodibenzofurans.

SIR REPORT. FILE - 462221 .SD RUN - 1 HRMID OCDD AND OCDF

UNCALIBRATED.

458 0 HRS 22 MINS 33 SECS 0.1032 0.1573 0.0000

460 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000

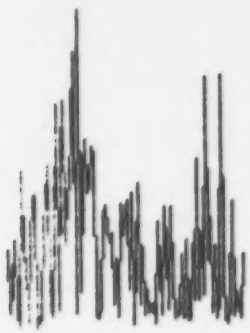
Test No. 1 - MM5 Front Sample
Column - 30m DB-5 Fused Silica

Figure B-9. HRGC/HRMS selected ion current profile for octachlorodibenzo-p-dioxin.

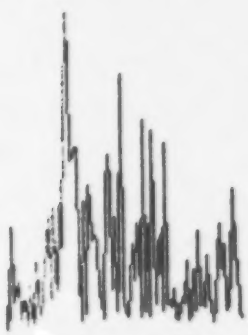
SIR REPORT. FILE - 462221 .SD RUN - 1 HRMID OCDD AND OCDF

UNCALIBRATED.

442 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000



444 0 HRS 22 MINS 34 SECS 0.0000 0.0000 0.0000



Reproduced from best available copy.

Test No. 1 - MM5 Front Sample
Column - 30m DB-5 Fused Silica

Figure B-10. HRGC/HRMS selected ion current profile for octachlorodibenzofuran.

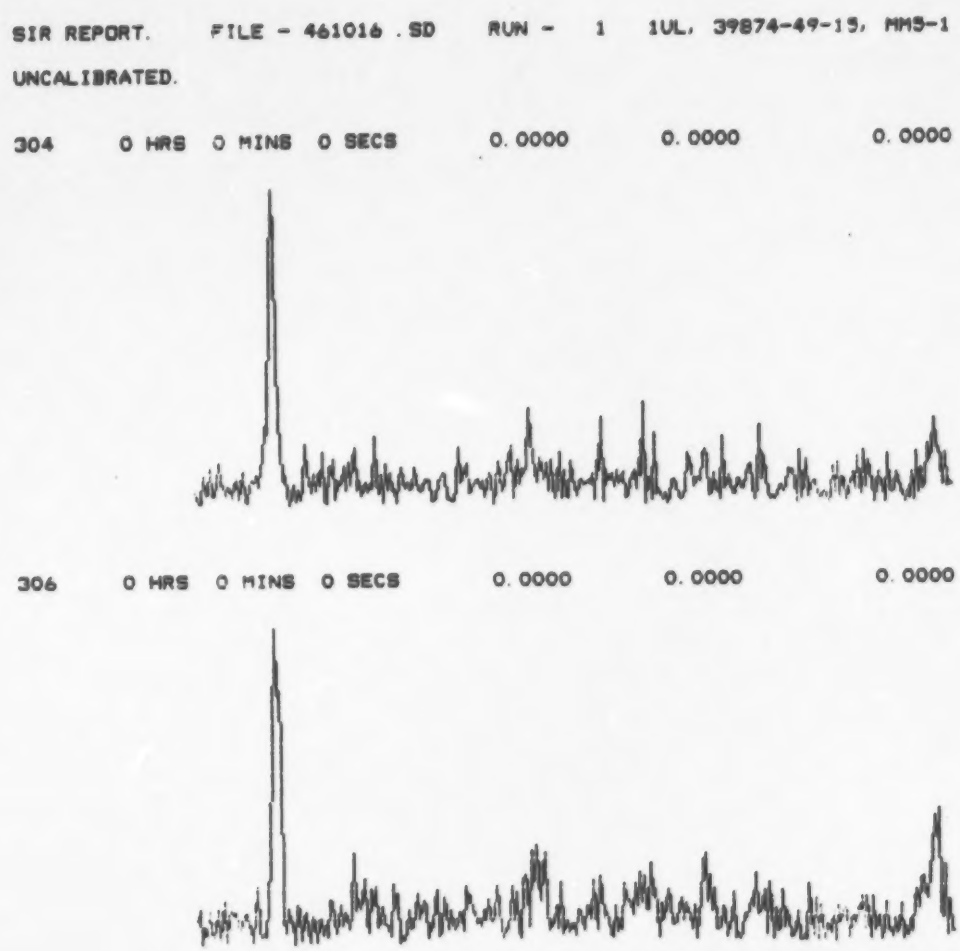

SIR REPORT. FILE - 461016 .SD RUN - 1 1UL, 39874-49-15, MM5-1
UNCALIBRATED.
304 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000
306 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000
Test No. 1 - MM5 Front Sample
Column - CP Sil-88

Figure B-11. HRGC/HRMS selected ion current profile for tetrachlorodibenzofurans.

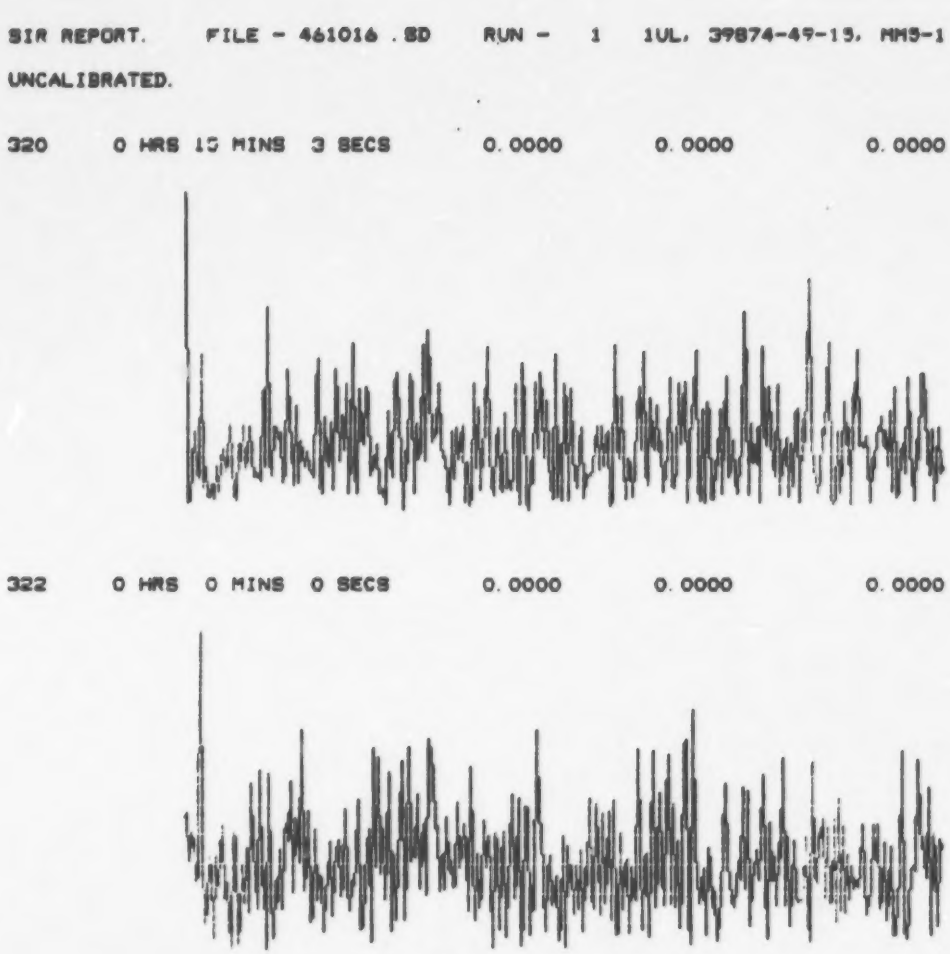

SIR REPORT. FILE - 461016 .SD RUN - 1 1UL. 39874-49-15. MM5-1
UNCALIBRATED.
320 0 HRS 15 MINS 3 SECS 0.0000 0.0000 0.0000
322 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000
Test NO. 1 - MM5 Front Sample
Column - CP Sil-88

Figure B-12. HRGC/HRMS selected ion current profile for tetrachlorodibenzo-p-dioxins.

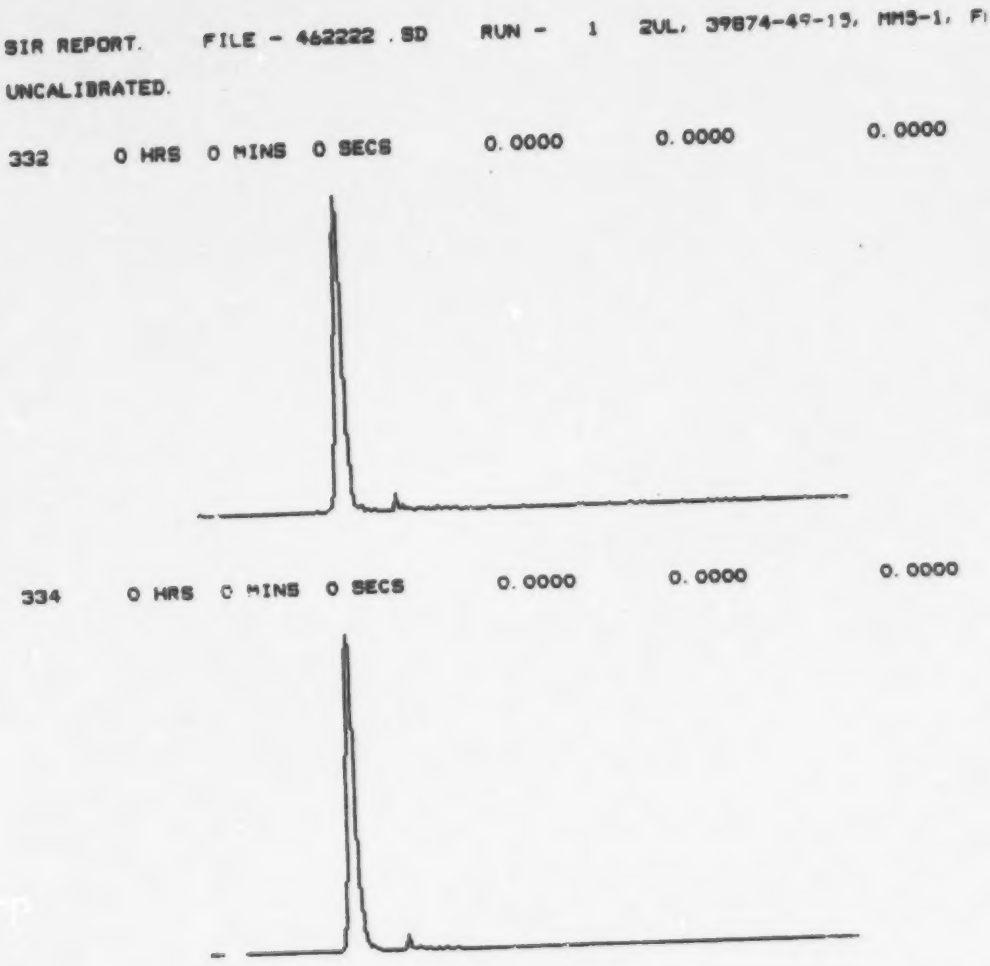
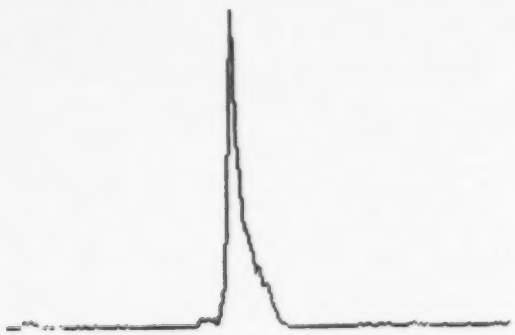

SIR REPORT. FILE - 462222 .SD RUN - 1 2UL, 39874-49-13, MM5-1, Fl
UNCALIBRATED.
332 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000
334 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000
Test No. 1 - MM5 Front Sample
Column - 30m DB-5 Fused Silica

Figure B-13. HRGC/HRMS selected ion current profile for 2,3,7,8-TCDD-$^{13}C_{12}$ internal standard.

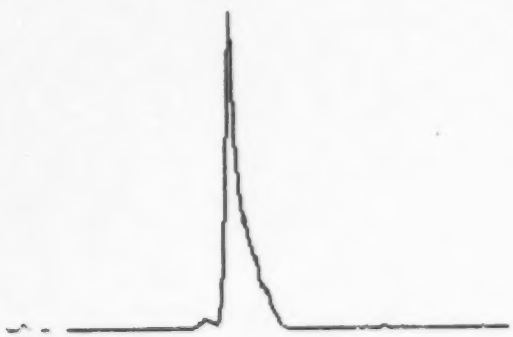
SIR REPORT. FILE - 462221 .SD RUN - 1 2UL, 39874-49-15, MM5-1, FI

UNCALIBRATED.

316 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000



318 0 HRS 7 MINS 3 SECS 0.0000 0.0000 0.0000



Test No. 1 - MM5 Front Sample
Column - 30m DB-5 Fused Silica

Figure B-14. HRGC/HRMS selected ion current profile for 2,3,7,8-TCDF-$^{13}C_{12}$ internal standard.

SIR REPORT. FILE - 462221 .SD RUN - 1 HRMID OCDD AND OCDF

UNCALIBRATED.

470	0 HRS 22 MINS 33 SECS	0.1640	0.2499	0.0000

472	0 HRS 22 MINS 33 SECS	0.0146	0.0222	0.0000

Test No. 1 - MM5 Front Sample
Column - 30m DB-5 Fused Silica

Figure B-15. HRGC/HRMS selected ion current profile for octachlorodibenzo-p-dioxin-$^{13}C_{12}$ internal standard.

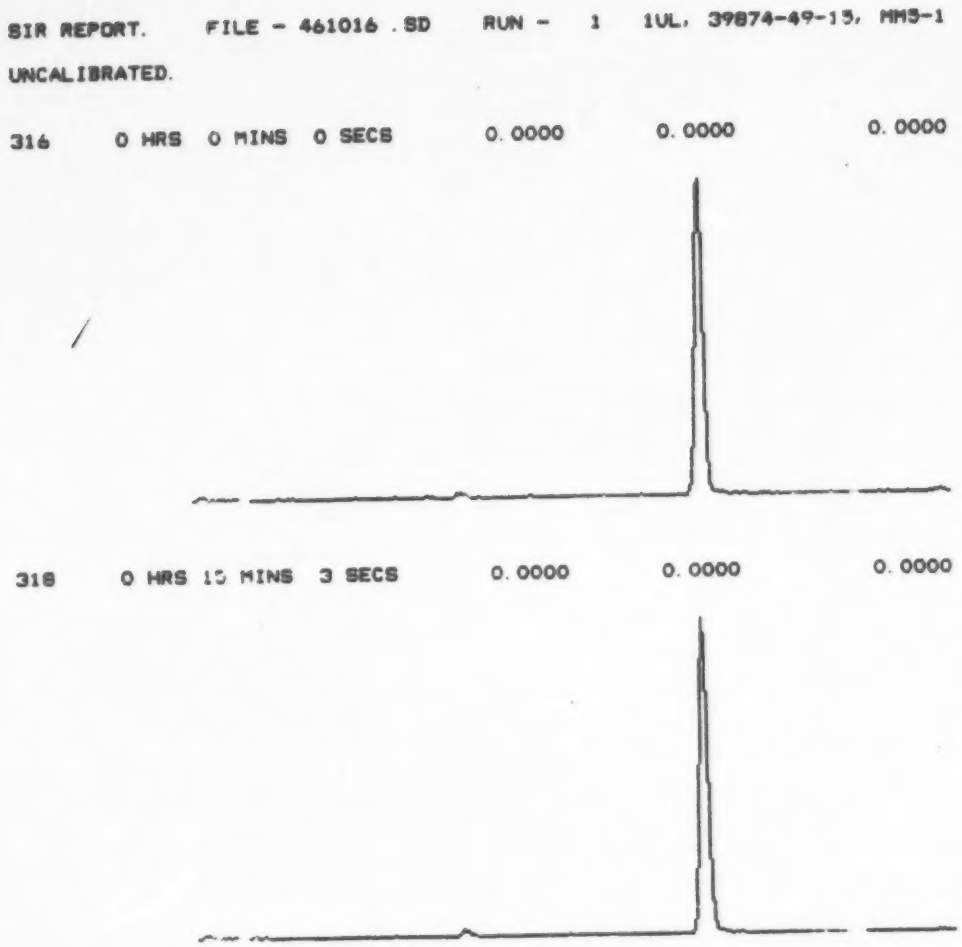

SIR REPORT. FILE - 461016 .SD RUN - 1 1UL, 39874-49-15, MM5-1
UNCALIBRATED.
316 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000
318 0 HRS 10 MINS 3 SECS 0.0000 0.0000 0.0000
Test No. 1 - MM5 Front Sample
Column - CP S11-88

Figure B-16. HRGC/HRMS selected ion current profile for 2,3,7,8-TCDF -$^{13}C_{12}$ internal standard.

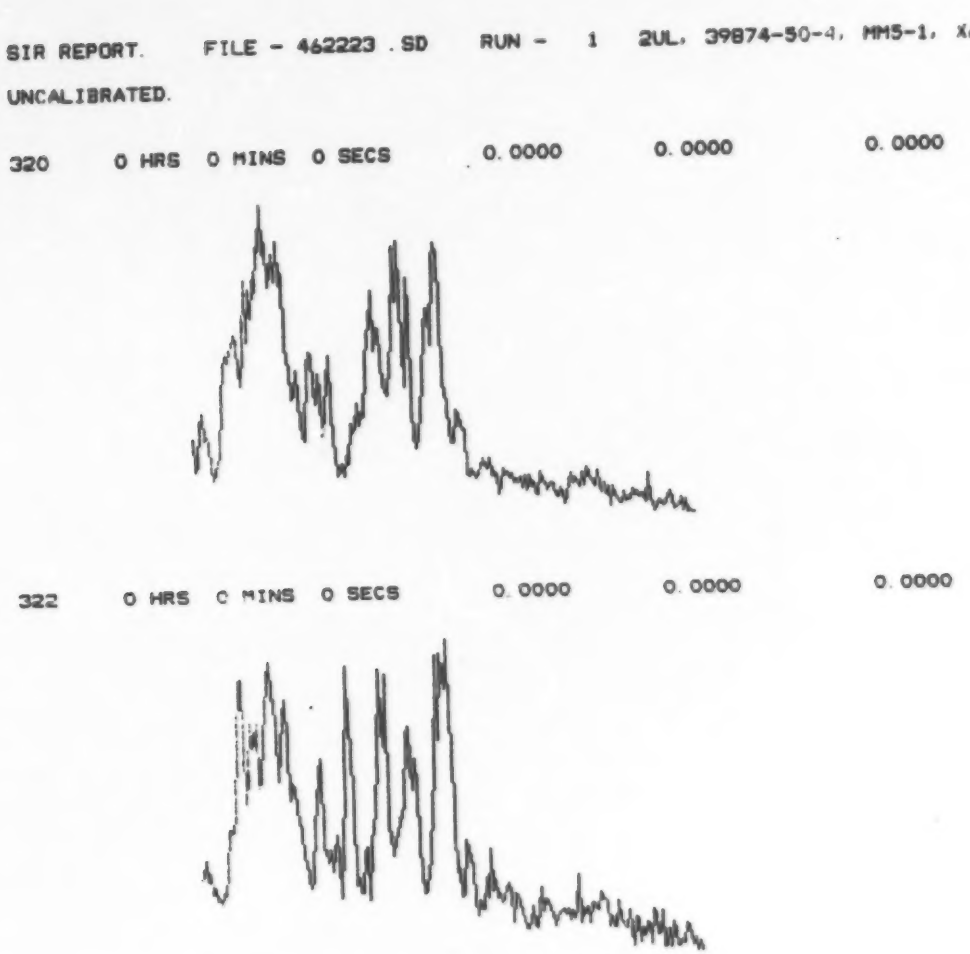

SIR REPORT. FILE - 462223 .SD RUN - 1 2UL, 39874-50-4, MM5-1, XA
UNCALIBRATED.
320 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000
322 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000
Test No. 1 - MM5 Back Sample
Column - 30m DB-5 Fused Silica

Figure B-17. HRGC/HRMS selected ion current profile for TCDDS.

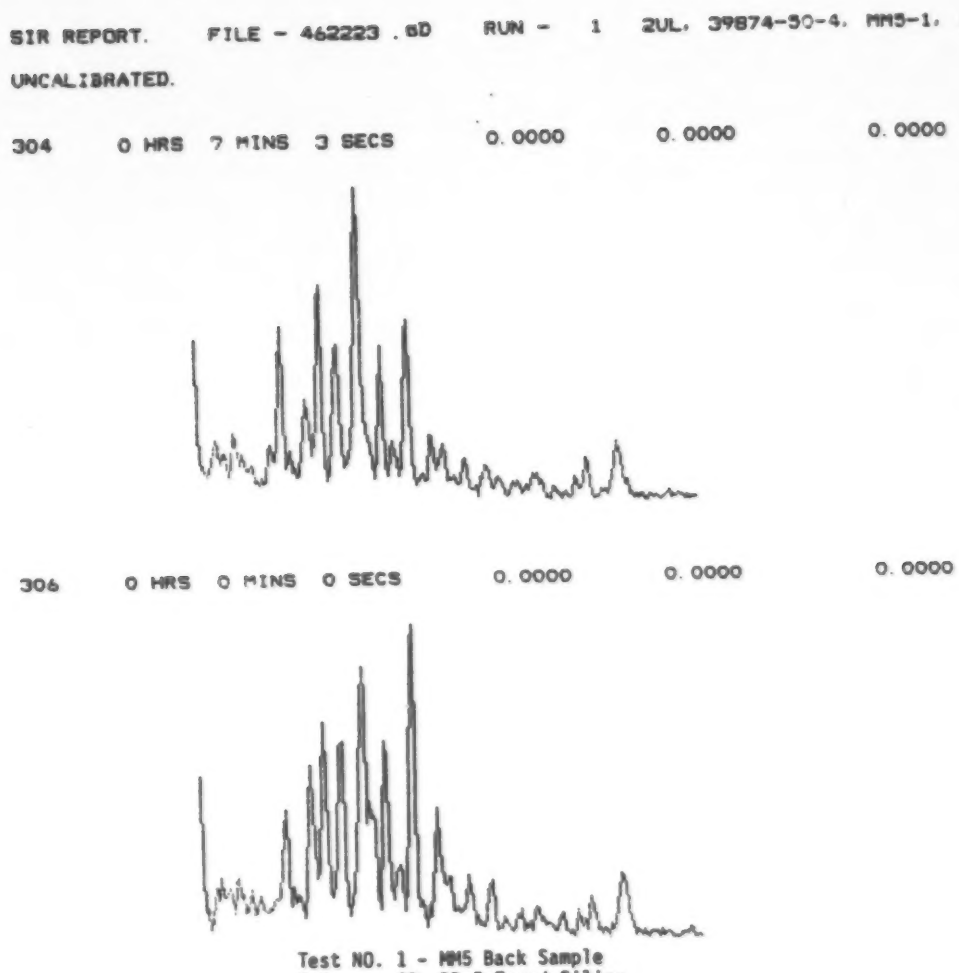

SIR REPORT. FILE - 462223 .6D RUN - 1 2UL, 39874-50-4, MM5-1, XA
UNCALIBRATED.
304 0 HRS 7 MINS 3 SECS 0.0000 0.0000 0.0000
306 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000
Test NO. 1 - MM5 Back Sample
Column - 30m DB-5 Fused Silica

Figure B-18. HRGC/HRMS selected ion current profile for TCDFs.

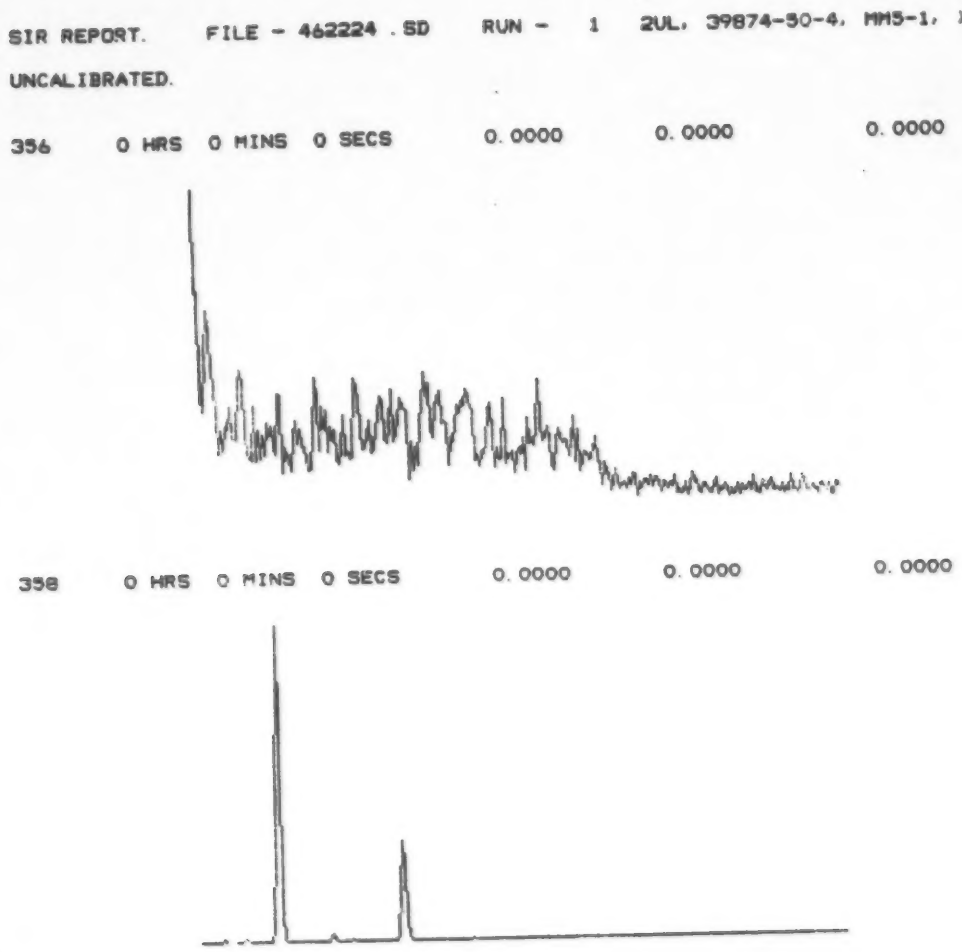
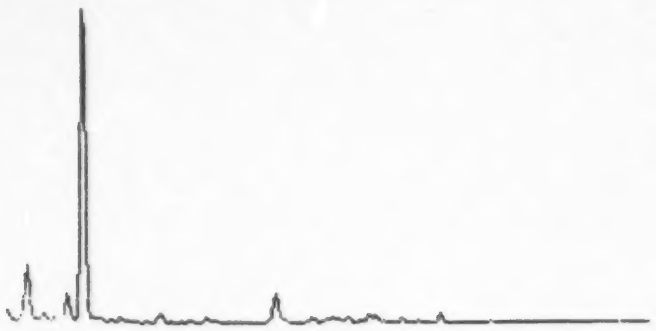

SIR REPORT. FILE - 462224 .SD RUN - 1 2UL, 39874-50-4, MM5-1, XA
UNCALIBRATED.
356 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000
358 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000
Test No. 1 - MM5 Back Sample
Column - 30m DB-5 Fused Silica

Figure B-19. HRGC/HRMS selected ion current profile for pentachlorodibenzo-p-dioxins.

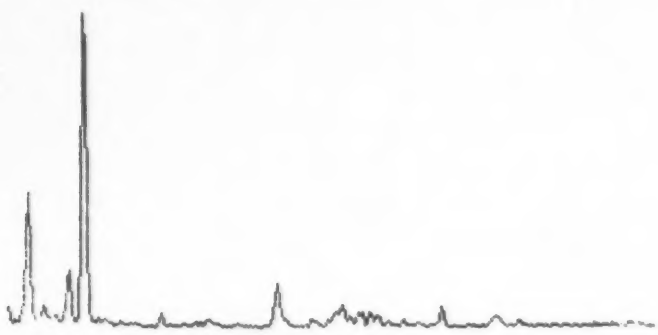
SIR REPORT. FILE - 462224 .SD RUN - 1 2UL, 39874-50-4, MM5-1, XA.

UNCALIBRATED.

340 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000



342 0 HRS 7 MINS 32 SECS 0.0000 0.0000 0.0000



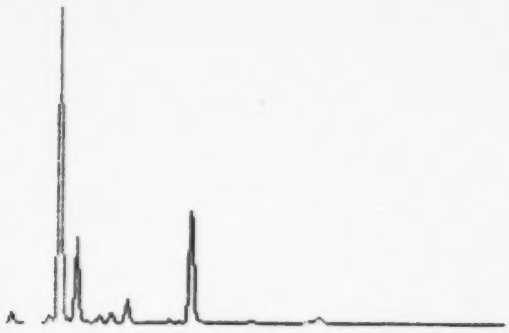
Test No. 1 - MM5 Back Sample
Column - 30m DB-5 Fused Silica

Figure B-20. HRGC/HRMS selected ion current profile for pentachlorodibenzofurans.

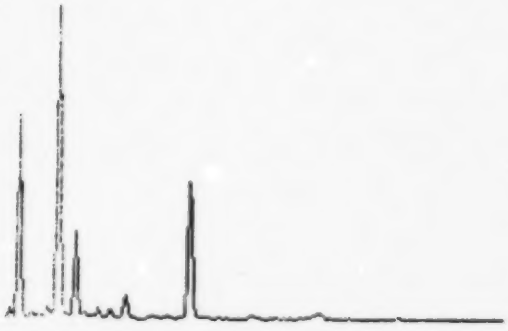
SIR REPORT. FILE - 462223 .SD RUN - 1 HRMID HXCDD AND HXCDF

UNCALIBRATED.

390 0 HRS 14 MINS 3 SECS 0.0000 0.0000 0.0000



392 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000



Test No. 1 - MM5 Back Sample
Column - 30m DB-5 Fused Silica

Figure B-21. HRGC/HRMS selected ion current profile for hexachlorodibenzo-p-dioxins.

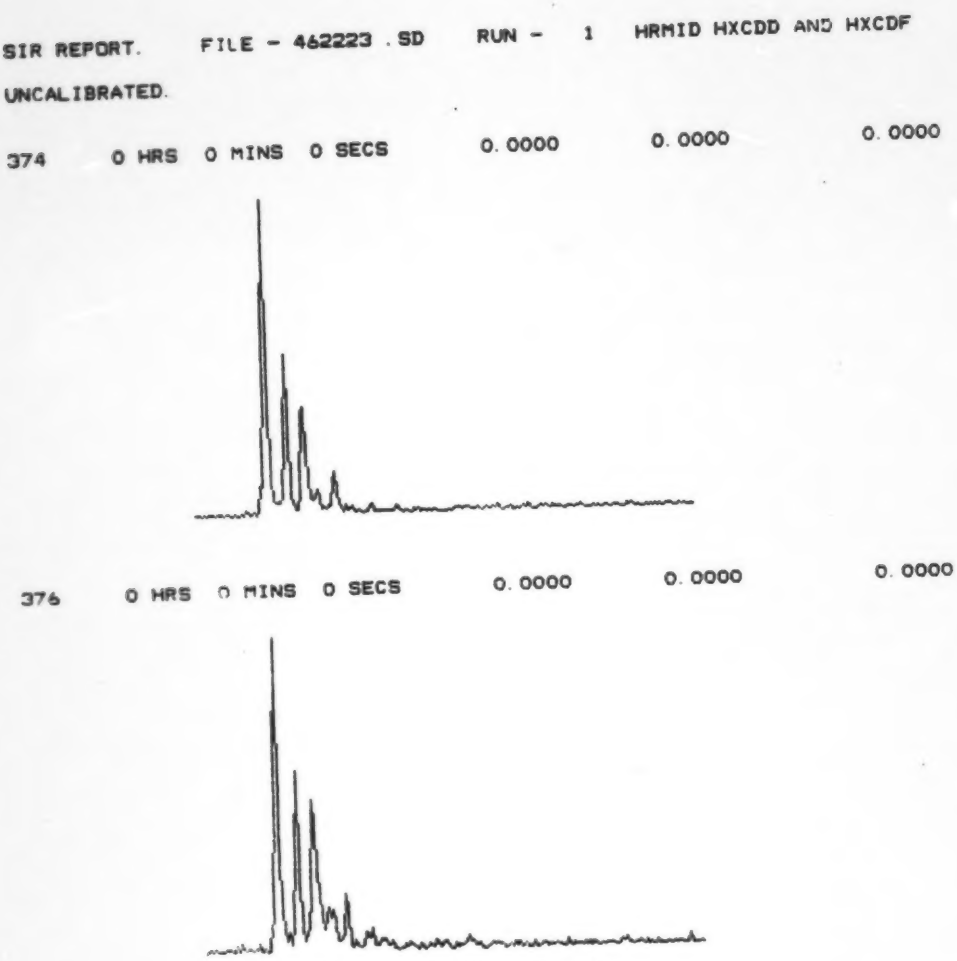

SIR REPORT. FILE - 462223 .SD RUN - 1 HRMID HXCDD AND HXCDF
UNCALIBRATED.
374 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000
376 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000
Test No. 1 - MM5 Back Sample
Column - 30m DB-5 Fused Silica

Figure B-22. HRGC/HRMS selected ion current profile for hexachlorodibenzofurans.

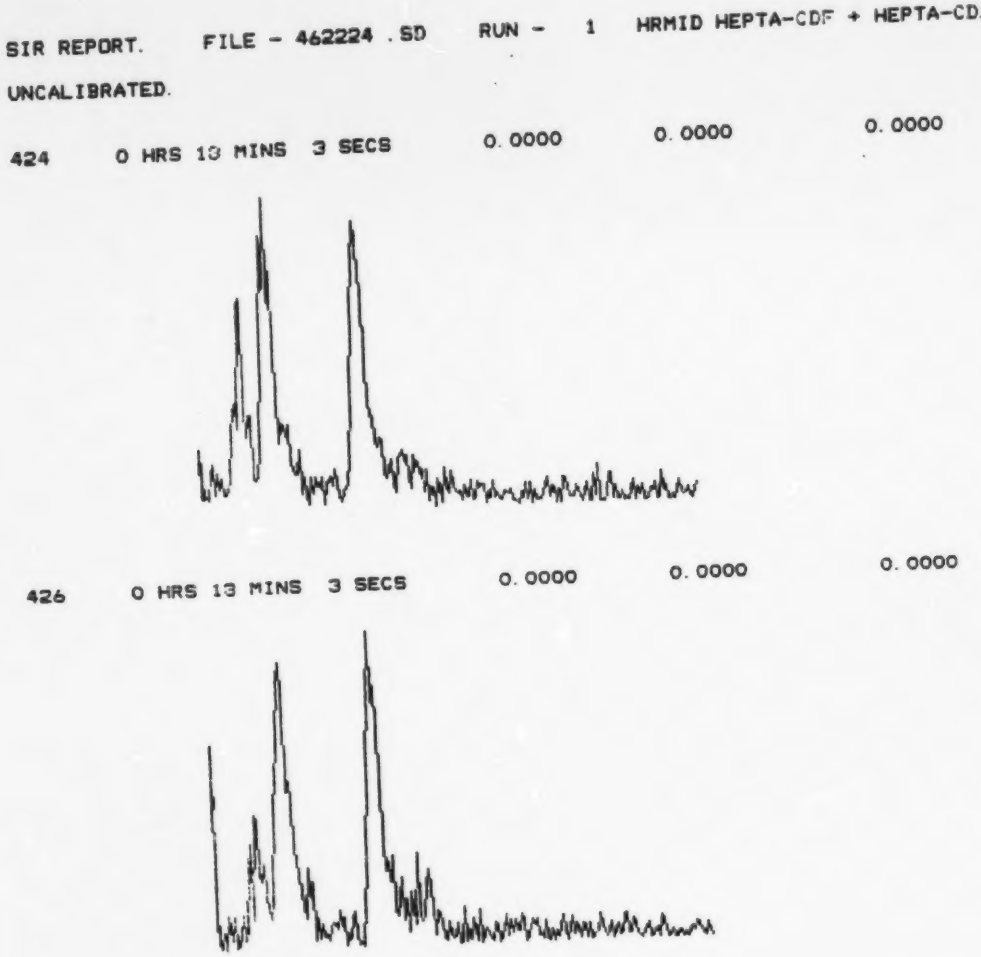
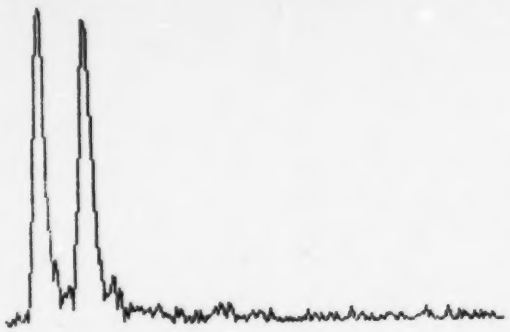

SIR REPORT. FILE - 462224 .SD RUN - 1 HRMID HEPTA-CDF + HEPTA-CD.
UNCALIBRATED.
424 0 HRS 13 MINS 3 SECS 0.0000 0.0000 0.0000
426 0 HRS 13 MINS 3 SECS 0.0000 0.0000 0.0000
Test No. 1 - MM5 Back Sample
Column - 30m DB-5 Fused Silica

Figure B-23. HRGC/HRMS selected ion current profile for heptachlorodibenzo-p-dioxins.

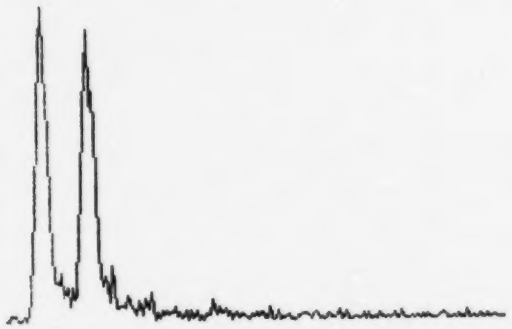
SIR REPORT. FILE - 462224 .SD RUN - 1 HRMID HEPTA-CDF + HEPTA-CD.

UNCALIBRATED.

408 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000



410 0 HRS 18 MINS 3 SECS 0.0000 0.0000 0.0000



Test No. 1 - MM5 Back Sample
Column - 30m DB-5 Fused Silica

Figure B-24. HRGC/HRMS selected ion current profile for heptachlorodibenzofurans.

SIR REPORT. FILE - 462223 .SD RUN - 1 HRMID OCDD AND OCDF

UNCALIBRATED.

458 0 HRS 22 MINS 33 SECS 0.1022 0.1558 0.0000

460 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000

Test NO. 1 - MM5 Back Sample
Column - 30m DB-5 Fused Silica

Figure B-25. HRGC/HRMS selected ion current profile for octachlorodibenzo--p-dioxin.

SIR REPORT. FILE - 462223 .SD RUN - 1 HRMID OCDD AND OCDF

UNCALIBRATED.

442 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000

444 0 HRS 22 MINS 33 SECS 0.0752 0.1146 0.0000

Test NO. 1 - MM5 Back Sample
Column - 30m DB-5 Fused Silica

Figure B-26. HRGC/HRMS selected ion current profile for octachlorodibenzofuran.

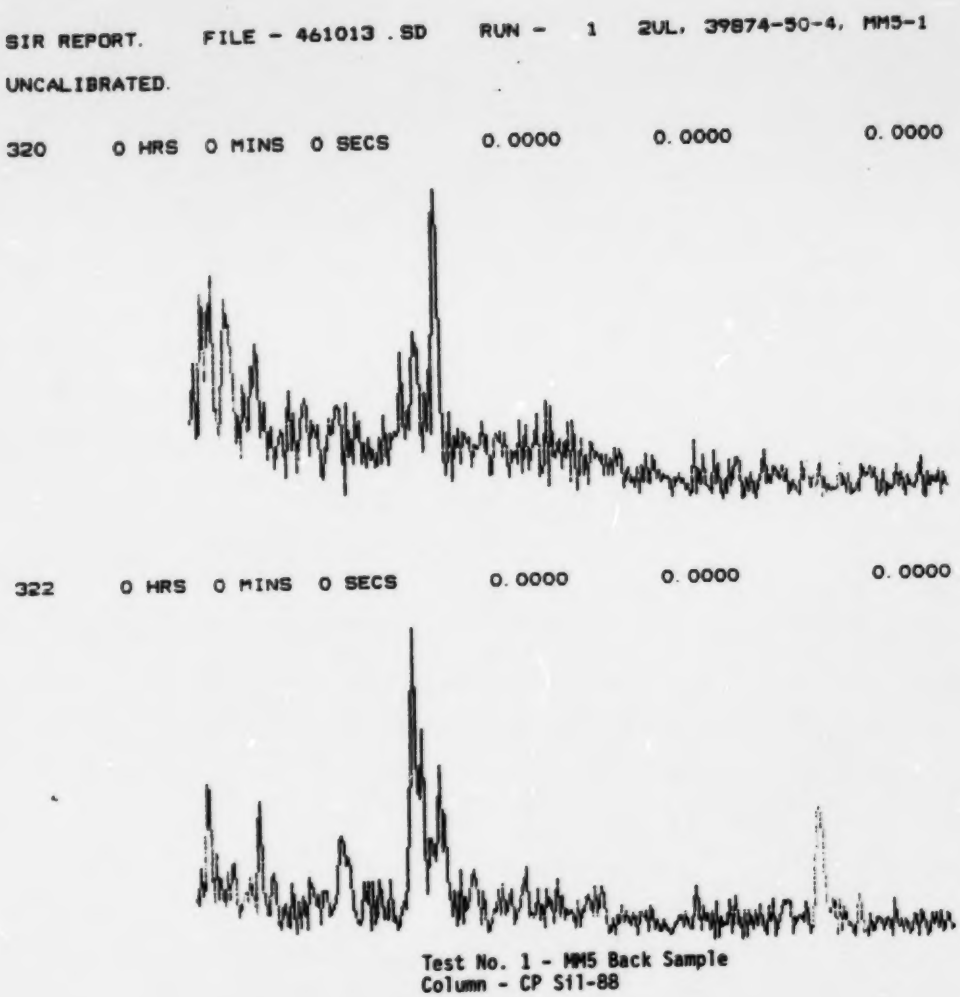

SIR REPORT. FILE - 461013 .SD RUN - 1 2UL, 39874-50-4, MM5-1
UNCALIBRATED.
320 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000
322 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000
Test No. 1 - MM5 Back Sample
Column - CP Sil-88

Figure B-27. HRGC/HRMS selected ion current profile for tetrachlorodibenzo-p-dioxins.

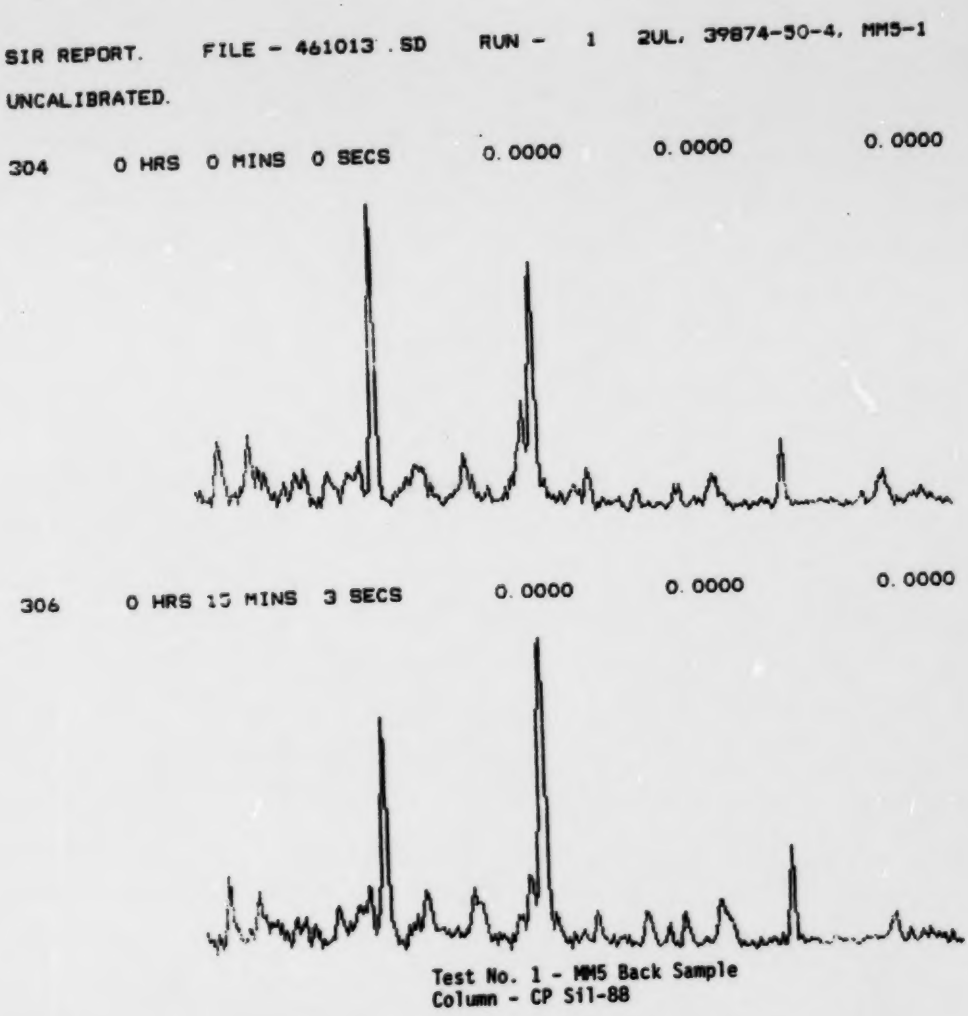
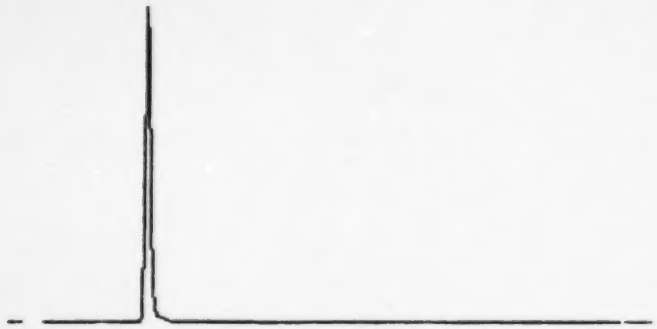

SIR REPORT. FILE - 461013 .SD RUN - 1 2UL, 39874-50-4, MM5-1
UNCALIBRATED.
304 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000
306 0 HRS 13 MINS 3 SECS 0.0000 0.0000 0.0000
Test No. 1 - MM5 Back Sample
Column - CP Sil-88

Figure B-28. HRGC/HRMS selected ion current profile for tetrachlorodibenzofurans.

SIR REPORT. FILE - 462224 .SD RUN - 1 2UL, 39674-50-4, MM5-1, XA

UNCALIBRATED.

332 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000



334 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000

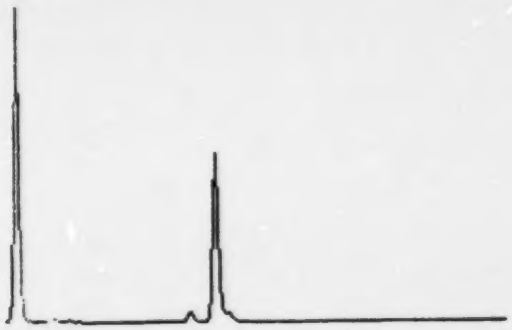
Test No. 1 - MM5 Back Sample
Column - 30m DB-5 Fused Silica

Figure B-29. HRGC/HRMS selected ion current profile for 2,3,7,8-TCDD-$^{13}C_{12}$

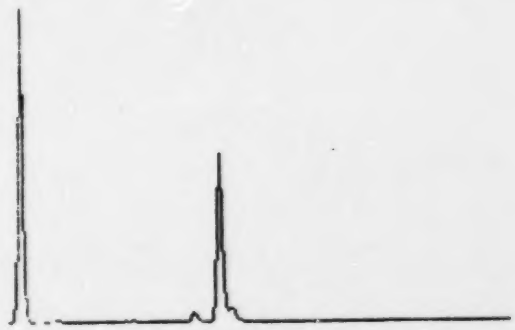
SIR REPORT. FILE - 462223 .SD RUN - 1 2UL, 39874-50-4, MM5-1, XA

UNCALIBRATED.

316 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000



318 0 HRS 7 MINS 3 SECS 0.0000 0.0000 0.0000



Test No. 1 - MM5 Back Sample
Column - 30m DB-5 Fused Silica

Figure B-30. HRGC/HRMS selected ion current profile for 2,3,7,8-TCDF-$^{13}C_{12}$ internal standard.

SIR REPORT. FILE - 462223 .SD RUN - 1 HRMID OCDD AND OCDF

UNCALIBRATED.

470	0 HRS 22 MINS 33 SECS	0.1308	0.1992	0.0000

472	0 HRS 22 MINS 33 SECS	0.0304	0.0463	0.0000

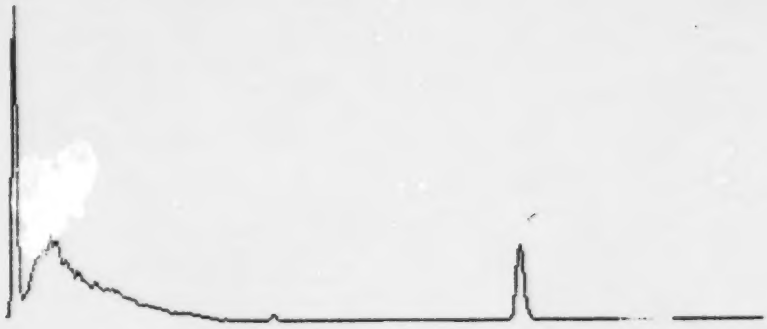
Test NO. 2 - MM5 Back Sample
Column - 30m DB-5 Fused Silica

Figure B-31. HRGC/HRMS selected ion current profile for octachlorodibenzo--p-dioxin-$^{13}C_{12}$ internal standard.

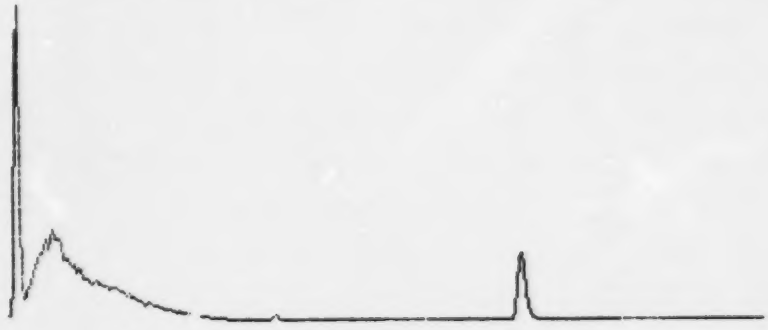
SIR REPORT. FILE - 461013 .SD RUN - 1 2UL, 39874-50-4, MM5-1

UNCALIBRATED.

316 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000



318 0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000



Test NO. 1 MM5 Back Sample
Column - CP Sil-88

Figure B-32. HRGC/HRMS selected ion current profile for 2,3,7,8-TCDF-$^{13}C_{12}$ internal standard.

APPENDIX C

CALIBRATION DATA

Calibration data for the dry gas meters in the modified Method 5 trains are given in Figures C-1 through C-4.

Calibration Date 11-29/84

Performed by Doyle Kohler

Barometer (P_b) 29.18 in. Hg

Sample Box S/N 1970

Dry Gas Meter S/N 946569

Wet Test Meter S/N _____

Orifice Manometer, in. H_2O (ΔH)	Gas Volumes, ft^3		Temperatures, F				Time, min. θ	Ratio V_w/V_d, Y	Dry Test $\Delta H@$, in. H_2O
	Wet Test (V_w)	Dry Gas (V_d)	Wet Test (t_w)	Dry Gas Inlet	Dry Gas Outlet	Dry Gas Avg. (t_d)			
0.5	5	I 880.800 F 885.700	80	80/90	79/81	82°F	12.76	1.0113	1.94
1.0	5	I 886.184 F 891.003	80	90/100	82/85	89°F	9.22	1.0424	2.00
2.0	10	I 891.390 F 901.276	80	98/108	86/90	96°F	13.10	1.0264	1.99
4.0	10	I 901.880 F 911.765	80	106/119	90/96	103°F	9.33	1.0345	2.00
		I _____ F							
		I _____ F							
							Average	1.029	1.98

Figure C-1. Method 5 dry gas meter pre-sampling calibration data.

Calibration Date 12/18/84

Performed by D. Kohler

Barometer (P_b) 29.56 in. Hg

Sample Box S/N 1970

Dry Gas Meter S/N 946569

Wet Test Meter S/N

Orifice Manometer, in. H_2O (ΔH)	Gas Volumes, ft^3		Temperatures, F				Time, min. θ	Ratio V_w/V_d, Y	Dry Test $\Delta H@$, in. H_2O
	Wet Test (V_w)	Dry Gas (V_d)	Wet Test (t_w)	Dry Gas Inlet	Dry Gas Outlet	Dry Gas Avg. (t_d)			
0.5	5	I 191.847 F 196.762	77	73/84	73/75	76	12.80	1.017	1.881
1.0	5	I 197.400 F 202.284	77	86/95	76/80	84	9.267	1.035	1.952
2.0	10	I 202.600 F 212.465	77	95/103	80/86	91	13.283	1.035	1.981
4.0	10	I 213.000 F 222.844	77	105/111	86/90	98	9.500	1.045	2.001
		I F							
		I F							
							Average	1.033	1.954

Figure C-2. Method 5 dry gas meter post-sampling data.

Calibration Date 11/30/84

Performed by Doyle Kohler

Barometer (P_b) 29.07 in. Hg

Sample Box S/N 1971

Dry Gas Meter S/N 946569

Wet Test Meter S/N

Orifice Manometer, in. H_2O (ΔH)	Gas Volumes, ft^3		Temperatures, F				Time, min. θ	Ratio V_w/V_d, Y	Dry Test $\Delta H@$, in. H_2O
	Wet Test (V_w)	Dry Gas (V_d)	Wet Test (t_w)	Dry Gas Inlet	Dry Gas Outlet	Dry Gas Avg. (t_d)			
0.5	5	I 738.500 F 743.373	76	68/78	70/74	72.5°F	12.483	1.010	1.867
1.0	5	I 743.662 F 748.541	76	79/86	74/78	79.3°F	8.90	1.018	1.876
2.0	10	I 748.947 F 758.781	76	86/99	79/85	87.3	13.183	1.022	2.028
4.0	10	I 759.185 F 769.070	76	95/105	85/89	93.5	9.50	1.025	2.082
		I F							
		I F							
							Average	1.019	1.963

Figure C-3. Method 5 dry gas meter pre-sampling calibration data.

Calibration Date 12/18/84

Performed by Doyle Kohler

Barometer (P_b) 29.56 in. Hg

Sample Box S/N 1971

Dry Gas Meter S/N 946569

Wet Test Meter S/N

Orifice Manometer, in. H_2O (ΔH)	Gas Volumes, ft^3		Temperatures, F				Time, min. θ	Ratio V_w/V_d, Y	Dry Test $\Delta H@$, in. H_2O
	Wet Test (V_w)	Dry Gas (V_d)	Wet Test (t_w)	Dry Gas Inlet	Dry Gas Outlet	Dry Gas Avg. (t_d)			
0.5	5	I 761.500 F 766.324	77	74/83	74/77	77	12.35	1.035	1.757
1.0	5	I 766.600 F 771.450	77	84/90	78/82	84	8.917	1.041	1.810
2.0	10	I 771.800 F 781.528	77	91/99	82/87	90	13.10	1.047	1.931
4.0	10	I 782.000 F 791.790	77	99/105	87/91	96	9.45	1.046	1.989
		I F							
		I F							
							Average	1.042	1.872

Figure C-4. Method 5 dry gas meter post-sampling data.